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PARTICLE IMPACT DAMAGE IN SILICON NITRIDE.(U)
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PARTICLE IMPACT DAMAGE IN SILICON NITRIDE

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Annual Report — Part III

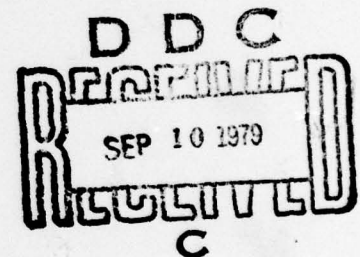
May 1979

By: K. C. Dao, D. A. Shockey, L. Seaman,
D. R. Curran, and D. J. Rowcliffe

Prepared for:

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SRI Project PYU-4928

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Annual Report, Part III 3,

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SUMMARY

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In the study of fracture in thick silicon nitride plates by particle impact and quasi-static indentation, we used three types of silicon nitride: hot pressed, reaction bonded, and oxidized hot pressed. The fractures were characterized, quantified, and where possible, correlated with calculated elastic stress fields.

→ The fracture damage ^{occurred} ~~was seen to occur~~ in well-defined progressive stages. For impact loads or quasi-static indentation loads that do not appreciably exceed the flow stress in the silicon nitride, the elastic radial tensile stresses cause ring and Hertzian cone cracks to form. In the dynamic case, the short duration of the tensile pulse restricts the formation of the ring cracks to the vicinity of the maximum contact radius of the impacting particle. In the quasi-static case, the ring cracks form well outside the maximum contact radius, suggesting that the fracture nucleation and growth kinetics play a significant role in determining the extent of damage.

For dynamic loads that exceed the flow stress of the silicon nitride, the plastic flow reduces the tensile radial stress and produces tensile hoop stresses, which in turn, cause radial cracks to form and propagate. At still higher dynamic loads, lateral cracks form. The intersection of these later-stage cracks with the free surface causes material removal (erosion). ←

Computer modeling with elastic-plastic material models will be required to improve our understanding of this fracture behavior. Radial and lateral cracks were not formed in the quasi-static indentation tests because flow and failure of the indentors prevented application of sufficient load levels.

The oxidized surface on hot pressed silicon nitride spalled locally under particle impact. Crack size determination showed that the oxide has little influence on the morphology and extent of fracture damage in the host material.

We found that the local crack propagation is sensitive to microstructural features in the hot pressed silicon nitride; the fractures were intergranular and the fracture paths were tortuous. Thus, resistance to erosion might be improved by modifying the size and shape of the grains. In reaction-bonded silicon nitride, on the other hand, the grains and pores are mostly smaller than 1 μm and do not appear to exert a marked influence on crack propagation and macrofracture.

At high temperatures representative of turbine blade operating conditions, the silicon nitride exhibited significantly more resistance to fracture, thus suggesting that future work should focus on this effect.

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I INTRODUCTION

Development of materials and fabrication processes for ceramic turbine components continues to emphasize silicon nitride and silicon carbide. Although hot pressed high-density silicon nitride has superior strength, the reaction bonded silicon nitride is more resistant to high-temperature creep and can be formed into components less expensively. Therefore, both types of silicon nitride hold promise of being useful gas turbine materials. However, because silicon nitride is brittle, it is very susceptible to localized surface cracking by repeated bombardments of debris and solid impurities in the hot and corrosive environment of a turbine engine. Brittle cracks that form in silicon nitride may cause serious strength degradation, leading to gradual deterioration in performance and even failure of the vital ceramic components. To solve this problem, we need to define the mechanisms that govern the impact erosion process in silicon nitride.

In our earlier work on zinc sulfide,¹ we noted the similarity between quasi-static indentation and dynamic particle impact fracture damage and suggested the use of quasi-static indentation experiments and theory to characterize the impact erosion process in ZnS. Silicon nitride, however, is a stronger ceramic and its response to particle contact situations is little known. Both quasi-static indentation and particle impact experiments were therefore required to determine if the quasi-static approach was adequate for silicon nitride.

The problem of progressive oxidation of silicon nitride at high turbine operating temperatures has been recently recognized,^{2,3} and it is believed that gradual thickening of the oxide scale coupled with significant spallation of the scale by particle impact may accelerate the erosion process. However, the effect of the oxide scale on the erosion process has not, to our knowledge, been experimentally investigated.

The Navy is also interested in determining whether microstructural features may influence resistance to impact erosion of these materials and, if so, how optimum microstructures can be produced.

The specific objectives of this research were to:

- (1) Define in detail the mechanism of particle impact fracture in hot pressed and reaction bonded silicon nitride.
- (2) Investigate the response of silicon nitride to quasi-static indentation.
- (3) Explain the effects of particle size, particle material, and the oxide scale on particle contact damage in silicon nitride.
- (4) Describe the influence of microstructural features on crack propagation and particle contact fracture damage.
- (5) Determine the effect of high temperature on impact erosion resistance of silicon nitride.
- (6) Develop analyses for predicting particle impact fracture behavior of silicon nitride.

In the remainder of this report we describe our progress toward meeting these objectives. Section II describes the material characterization, Sections III and IV discuss the dynamic and static experimentation, respectively, and Section V covers the observed effect of high temperature on the dynamic results. Section VI describes the observed correlation of fracture behavior with microstructural features, and Section VII discusses the analysis of the experimental data. Finally, in Section VIII we present our conclusions.

II SPECIMEN MATERIALS

Hot Pressed Silicon Nitride

Hot pressed silicon nitride (HP Si_3N_4) NC132 with a density of 3.2 g/cm^3 was obtained from Norton Company in the form of a flat billet approximately 15 cm by 15 cm by 1.3 cm. X-ray diffraction showed $\beta\text{-Si}_3\text{N}_4$ and small peaks attributed to WSi_2 and Si_2ON_2 . The WSi_2 results from reaction of tungsten carbide (WC) and Si_3N_4 during hot pressing, and the WC pick-up arises from the use of WC milling balls in the original powder processing. Specimens for the impact experiments were all taken parallel to the hot pressing direction and polished to a $1\text{-}\mu\text{m}$ particle finish. In this condition the surface was essentially featureless. The microstructure illustrated in Figure 1 was revealed by etching in a solution of 30% HF, 25% H_2O_2 and 5% HNO_3 at 100°C for up to 2 minutes. The structure consists of $\beta\text{-Si}_3\text{N}_4$ with large grains of about $2 \mu\text{m}$ and submicrometer grains. Most grains are equiaxed, but occasional ones have aspect ratios up to 10:1. No microstructural evidence of grain boundary phases or inclusions could be found.

Oxide Scale

Several samples of NC132 were oxidized for 100 hours at 1250°C in air to form an oxide scale about $20 \mu\text{m}$ thick. The structure and composition of scales on NC132 have been studied in detail.^{2,3} Scales formed at 1250°C consisted of $\alpha\text{-cristobalite}$ and MgSiO_3 as major crystalline phases with small amounts of $\beta\text{-Si}_3\text{N}_4$ and Si_3ON_2 . The most striking microstructural feature of the scale was its porosity. Pores up to $5 \mu\text{m}$ in diameter were present throughout the scale's thickness, and pits were formed beneath the scale in the unoxidized material. The outer surface of the scale consisted of a thin layer of much less porous oxide.

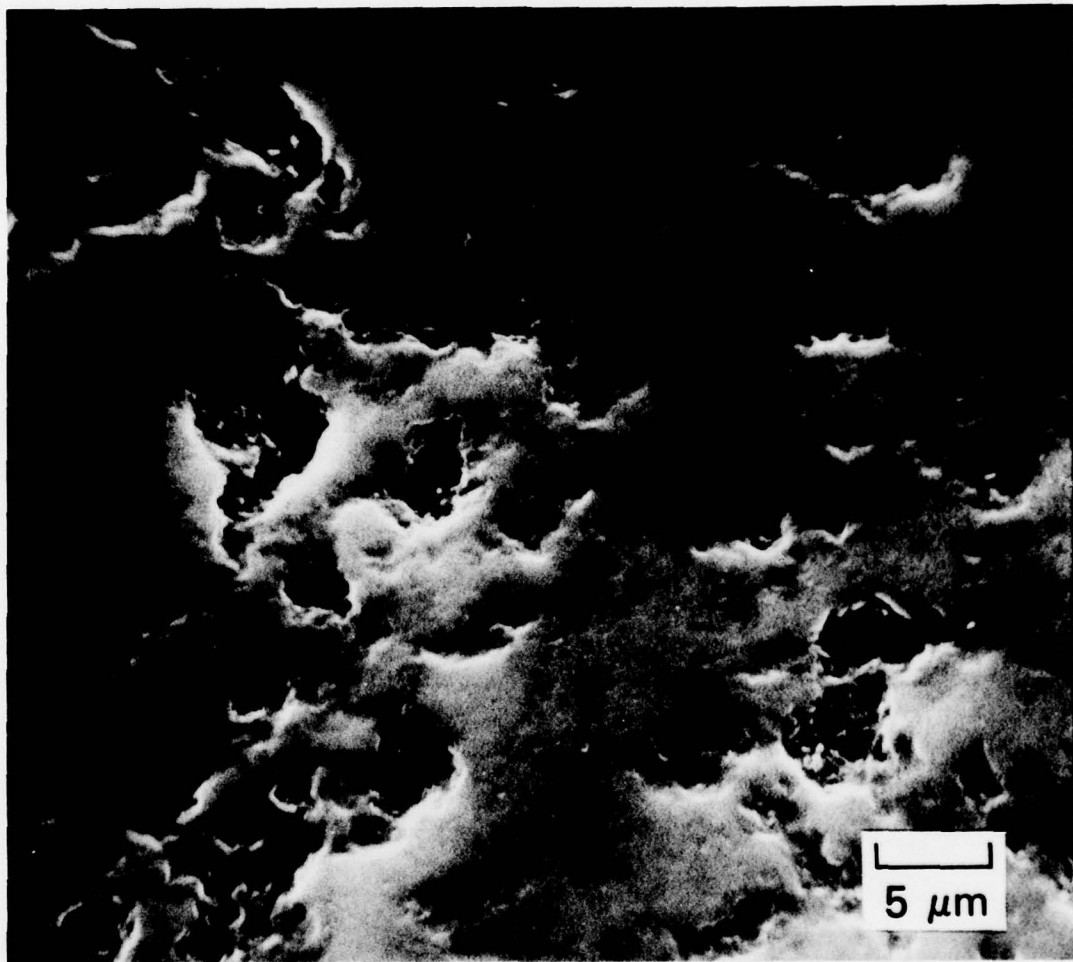


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FIGURE 1 MICROSTRUCTURE OF HOT PRESSED SILICON NITRIDE

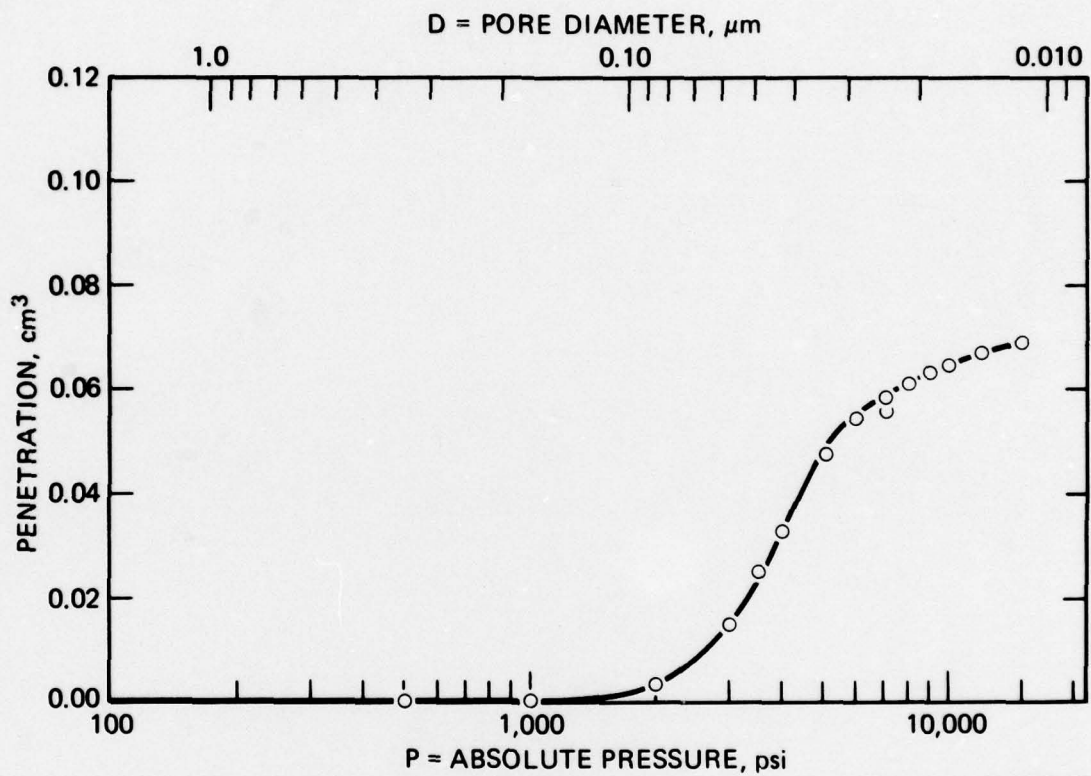
Reaction Bonded Silicon Nitride

Reaction bonded silicon nitride (RB Si_3N_4), NC350, was obtained from the Norton Company in the form of a disc approximately 22.5 cm in diameter by 6 cm thick. Test specimens were polished to a 1- μm particle finish. A scanning electron micrograph of the microstructure at the specimen surface is shown in Figure 2. Density and pore size distribution were measured using a mercury porosimeter; the results are shown in Figure 3. The density was 2.65 g/cm^3 , which is 84.5% of the theoretical value. The pore size ranged from 0.01 to 0.1 μm , with the mean close to 0.05 μm . X-ray diffraction studies identified α - and β - Si_3N_4 with no other second phases. A comparison of peak intensities showed the material to consist of approximately 60% α - Si_3N_4 and 40% β - Si_3N_4 .



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FIGURE 2 MICROSTRUCTURE OF REACTION BONDED SILICON NITRIDE



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FIGURE 3 PORE SIZE DISTRIBUTION IN REACTION BONDED SILICON NITRIDE (PENETRATION IS A DIRECT MEASURE OF VOLUME FRACTION OF PORES AND MAXIMUM PENETRATION, ABOUT 0.07 cm^3 CORRESPONDS TO VOLUME FRACTION OF ALL PORES, 0.155)

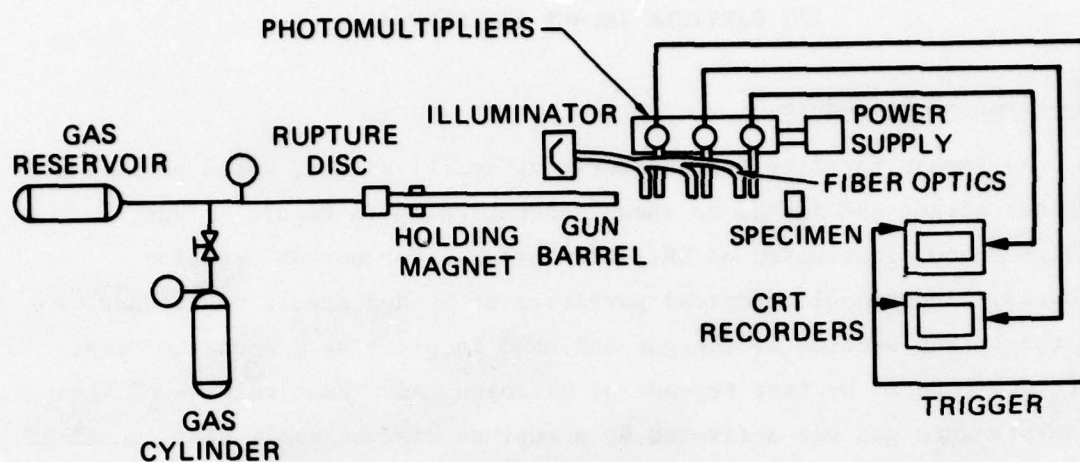
III PARTICLE IMPACT EXPERIMENTS

Experimental Procedure

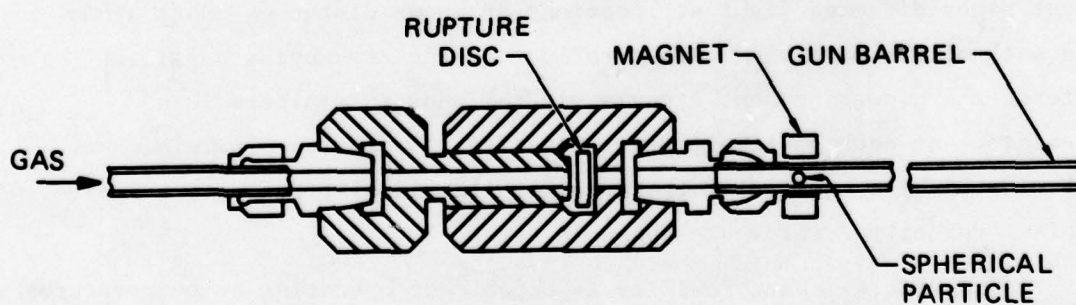
An impact facility for accelerating small, single, solid particles against target specimens, as shown schematically in Figure 4, was designed and constructed at SRI International for our ONR erosion program. Individual spherical particles of WC and steel, positioned in the breech section of the gun and held in place by a magnetic ring, were accelerated by fast release of nitrogen gas. Fast release of the high-pressure gas was activated by a rupture disc assembly that consisted essentially of a thin disc located in a two-part chamber, Figure 4(b). The gas pressure was allowed to build up on one side of the chamber. At a desired pressure, the disc ruptured, dumping the gas to the side where the particle was initially at rest. The expansion of the gas was rapid and the particle was propelled to high velocities. The disc material and thickness were varied to provide a wide range of velocity.

The impact and rebound velocities were measured by a system of three photomultipliers, each consisting of a photosensitive element located in a housing that was connected to a light pipe. The three light pipes directed light at locations of known distances apart along the path of the particle. Light reflected from the moving particle entered the pipes and sent signals via the photomultipliers to a recorder. At each photomultiplier location, the moving particle was illuminated by a common light source via individual fiber optics cables. Typical velocity records are shown in Figure 5.

The particle-impact facility is capable of operating at temperatures from ambient to 1500°C and at velocities from 13 to 700 m/s. Maximum impact velocity depends on particle size. Velocities higher than 700 m/s can be obtained by higher gas pressures and improved gas-release devices. Particles of the following diameters can be accelerated in the facility:



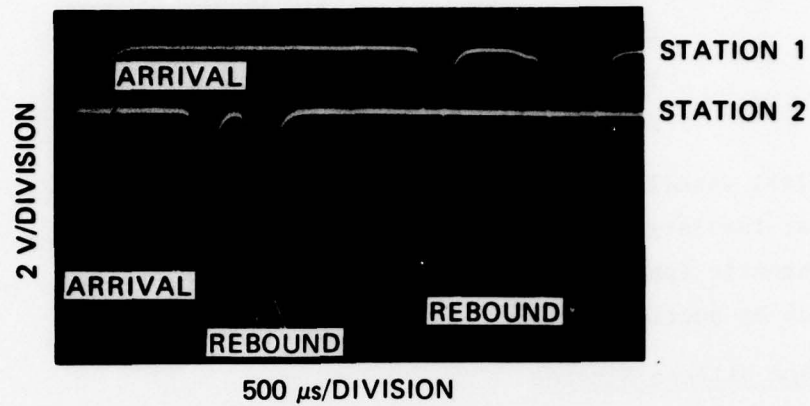
(a) SCHEMATIC OF THE FACILITY



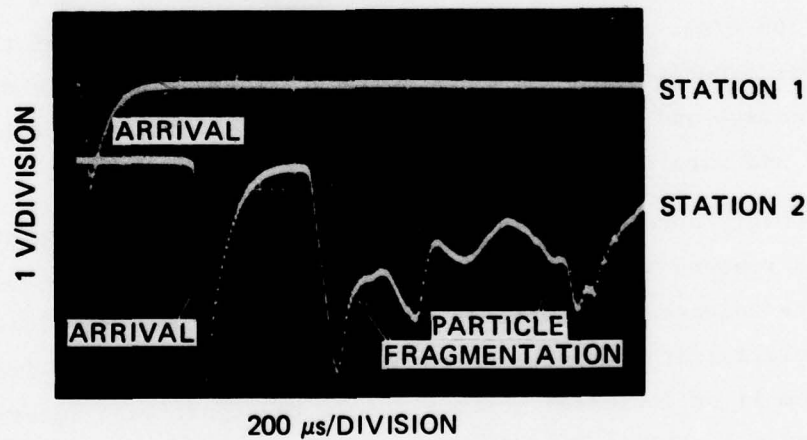
(b) DETAIL OF RUPTURE DISC ASSEMBLY FOR FAST RELEASE OF GAS

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FIGURE 4 SRI PARTICLE-IMPACT FACILITY



Particle: Steel, 2.4 mm
 Target: Si_3N_4
 Velocity: 56.4 m/s



Particle: WC, 2.4 mm
 Target: Si_3N_4
 Velocity: 120.9 m/s

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FIGURE 5 TYPICAL VELOCITY RECORDS

	<u>Diameter (mm)</u>
Tungsten carbide	0.4 to 4.8
Steel	1.0 to 4.8
Glass	0.1 to 4.8
Nylon	1.0 to 4.8
Al ₂ O ₃	3.2 to 4.8

The smallest diameter is dictated by the smallest commercially available particles; the largest is the diameter of the largest current gun barrel. The nonmagnetic spheres, glass, nylon and Al₂O₃, can be held in the gun breech by suction.

Hot Pressed Silicon Nitride

Summary of Particle Impact Experiments

We performed 43 particle impact experiments on HP Si₃N₄ using spherical particles of two sizes (1.6 and 2.4 mm diameter), two particle materials (tungsten carbide and steel), and a range of impact velocity (16.9 to 368 m/s). Table 1 lists the important parameters of these tests. The experiments were expected to provide a wide range of fracture damage and show influences of impact velocity, particle size, material, and particle/target combination.

Reasonably normal impacts and accurate velocity measurements (both impact and rebound velocities) were realized for most of the 43 experiments. The selected combinations of impact velocities, particle sizes, and particle/target combinations resulted in a wide range of fracture damage from light incipient surface cracks to significant material removal and target failure. Some particles were recovered and examined for impact damage. The results were included in Table 1.

Fractographic Observations

Optical micrographs of typical surface damage observed in the HP Si₃N₄ specimens are presented in Figures 6 through 8. The results show increasing surface damage with increased impact velocity as expected.

Table 1

PARTICLE IMPACT EXPERIMENTS ON HOT PRESSED SILICON NITRIDE

<u>Test No.</u>	<u>SRI No.</u>	<u>Impact Velocity (m/s)</u>	<u>Rebound Velocity (m/s)</u>	<u>Damage to Particle</u>
Impact configuration A (2.40-mm-diameter steel, HP Si ₃ N ₄)				
1	4928-119	42.3	a	Intact
2	4928-131	42.3	32	Intact
3	4928-133	46.2	a	Flattened
4	4928-132	56.4	34	Flattened
5	4928-129	62	36	b
6	4928-120	87.6	a	b
7	4928-121	105.8	52	b
8	4928-122	133.7	58	b
9	4928-124	141	63	b
10	4928-123	154	67	b
11	4928-126	195	69	b
12	4928-127	231	70	Fragmentation ^c
13	4928-128	299	a	Fragmentation ^c
Impact configuration B (2.40 mm-diameter WC, HP Si ₃ N ₄)				
14	4928-117	16.9	15	Intact
15	4928-116	19.5	16	Intact
16	4928-115	24.2	18	Flattened
17	4928-114	31.7	24	Flattened

^aRebound velocity not available or particle fragmented.

^bParticles were not recovered or no signals from fragments.

^cDetermined from photomultiplier signals of rebounded fragments.

Table 1 (continued)

PARTICLE IMPACT EXPERIMENTS ON HOT PRESSED SILICON NITRIDE

<u>Test No.</u>	<u>SRI No.</u>	<u>Impact Velocity (m/s)</u>	<u>Rebound Velocity (m/s)</u>	<u>Damage to Particle</u>
Impact configuration B (continued) (2.40 mm-diameter WC, HP Si ₃ N ₄)				
18	4928-112	33.8	25	Flattened
19	4928-113	39	27	b
20	4928-118	39	30	b
21	4928-111	46.2	35	b
22	4928-109	50.8	39	b
23	4928-110	52.9	39	b
24	4928-108	92.3	25	Fragmentation ^c
25	4928-107	97.7	a	Fragmentation ^c
26	4928-105	121	a	Fragmentation ^c
27	4928-106	121	a	Fragmentation ^c
28	4928-103	159	a	Fragmentation ^c
29	4928-102	169	a	Fragmentation ^c
30	4928-104	231	a	Fragmentation ^c
Impact configuration C (1.60-mm-diameter WC, HP Si ₃ N ₄)				
31	4928-99	37.6	30	b
32	4928-97	46.2	35	b
33	4928-96	67.7	43	Flattened
34	4928-94	78.1	a	Fragmentation ^c

^aRebound velocity not available or particle fragmented.

^bParticles were not recovered or no signals from fragments.

^cDetermined from photomultiplier signals or rebounded fragments.

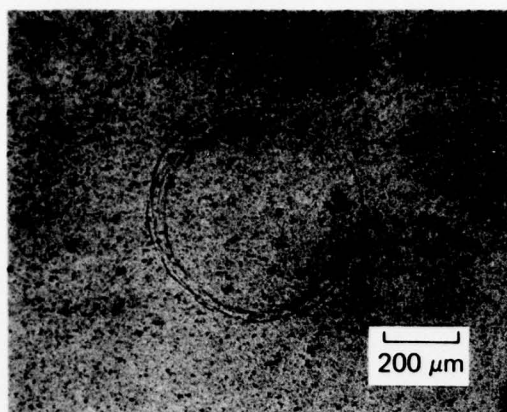
Table 1 (concluded)

<u>Test No.</u>	<u>SRI No.</u>	<u>Impact Velocity (m/s)</u>	<u>Rebound Velocity (m/s)</u>	<u>Damage to Particle</u>
Impact configuration C (concluded) (1.60-mm-diameter WC, HP Si ₃ N ₄)				
35	4928-95	78.1	30	Fragmentation ^c
36	4928-93	84.6	30	Fragmentation ^c
37	4928-92	92.4	24	Fragmentation ^c
38	4928-89	102	24	Fragmentation ^c
39	4928-91	127	a	Fragmentation ^c
40	4928-87	195	a	b
41	4928-100	254	a	Fragmentation ^c
42	4928-101	254	a	Fragmentation ^c
43	4928-64	368	a	b

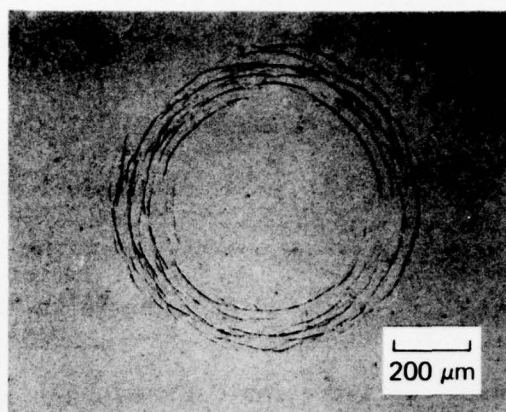
^aRebound velocity not available or particle fragmented.

^bParticles were not recovered or no signals from fragments.

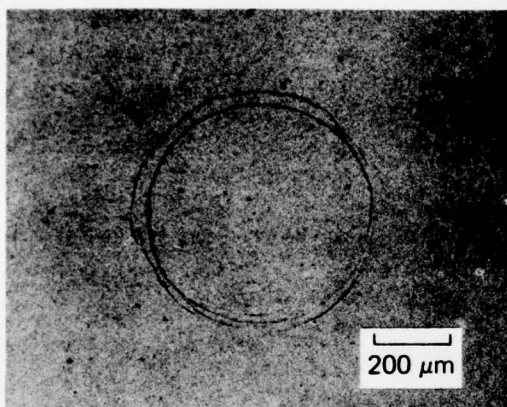
^cDetermined from photomultiplier signals of rebounded fragments.



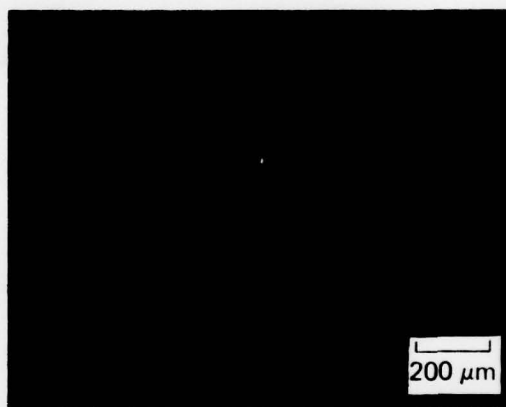
(a) 42.3 m/s



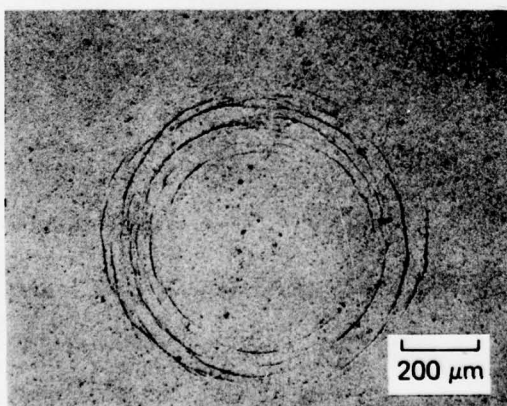
(d) 195 m/s



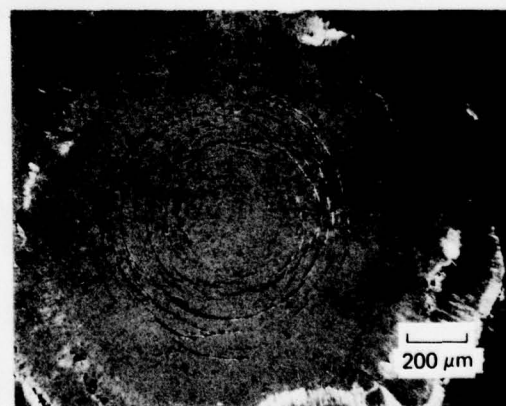
(b) 56.4 m/s



(e) 231 m/s



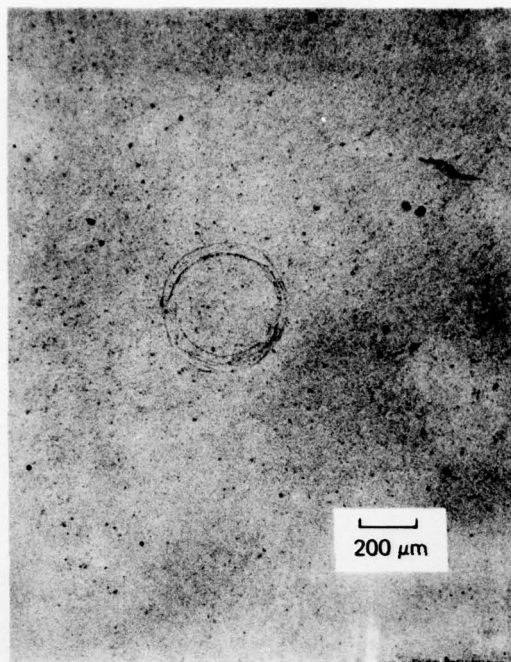
(c) 154 m/s



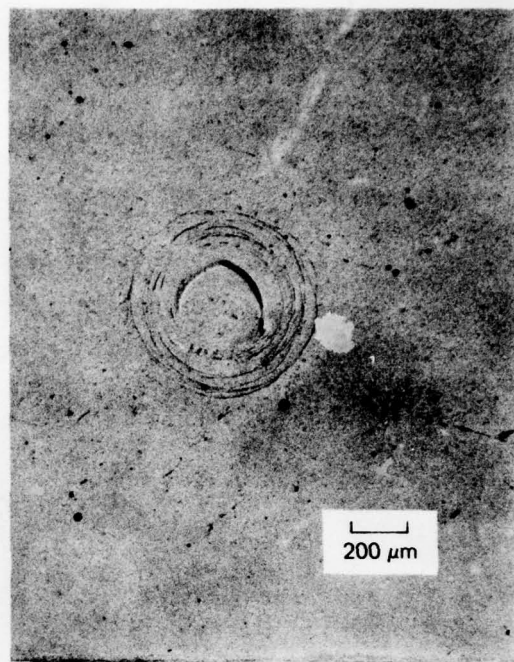
(f) 299 m/s

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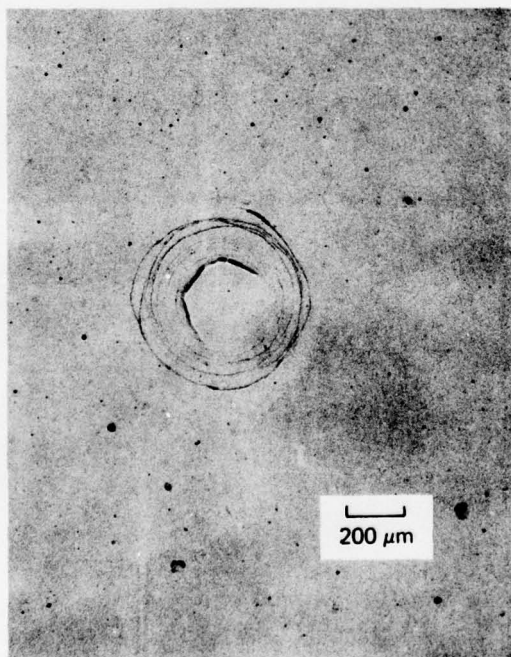
FIGURE 6 TYPICAL IMPACT DAMAGE IN HP Si_3N_4 BY 2.4-mm-DIAMETER STEEL SPHERES (Impact Configuration A)



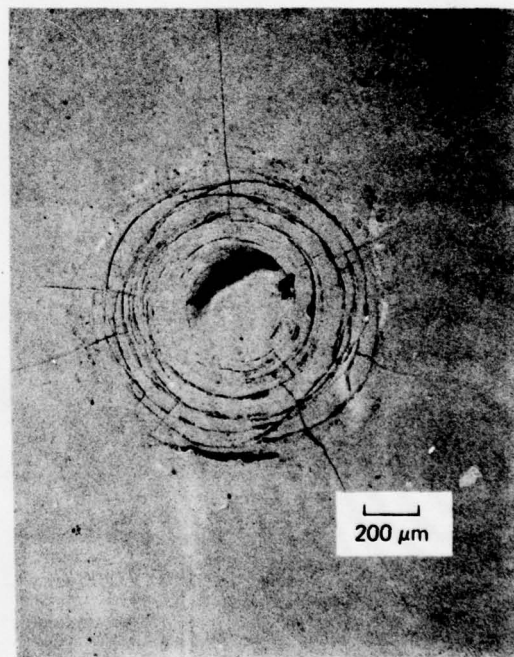
(a) 19.5 m/s



(c) 46.2 m/s



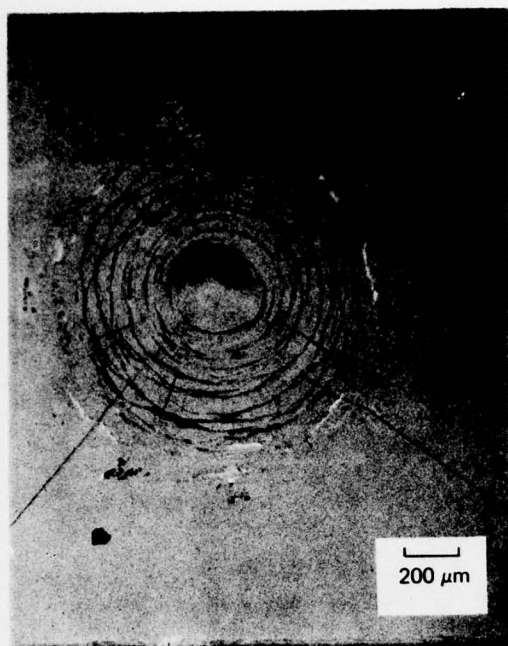
(b) 39 m/s



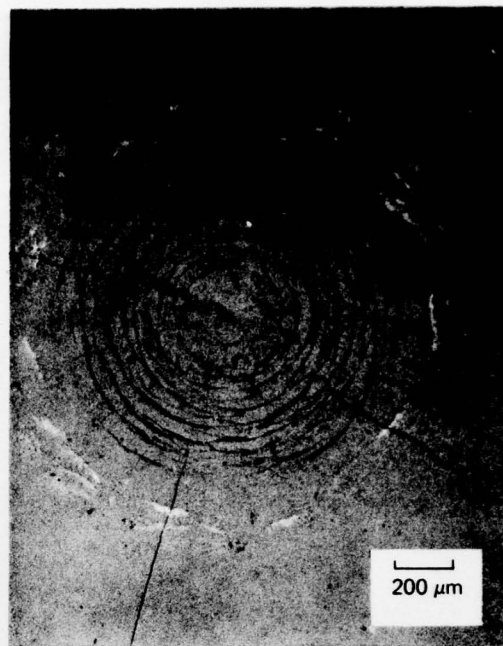
(d) 97.7 m/s

MP-4928-33

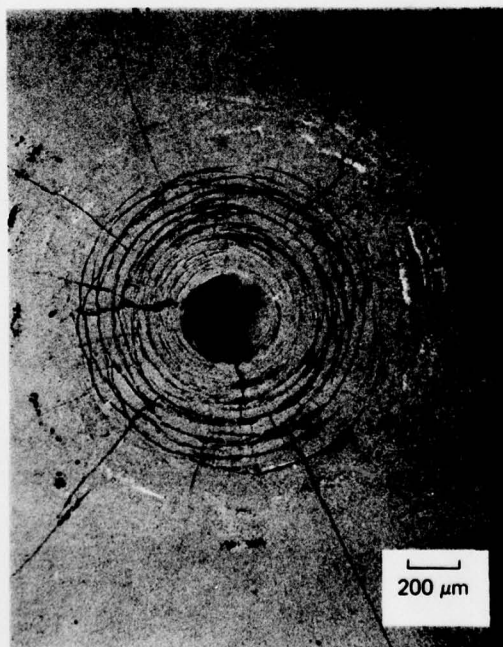
FIGURE 7 TYPICAL IMPACT DAMAGE IN HP Si_3N_4 BY 2.4-mm-DIAMETER WC SPHERES
(Impact Configuration B)



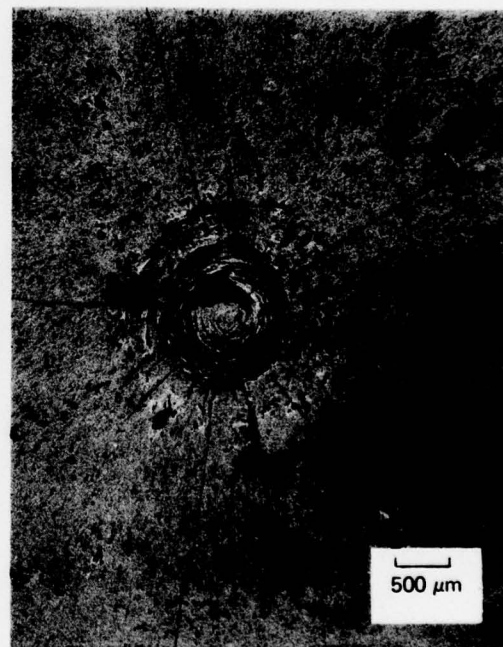
(e) 121 m/s



(g) 169 m/s



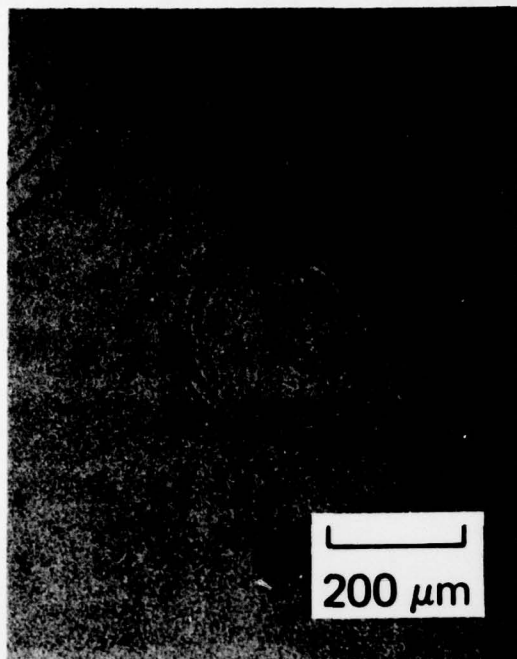
(f) 159 m/s



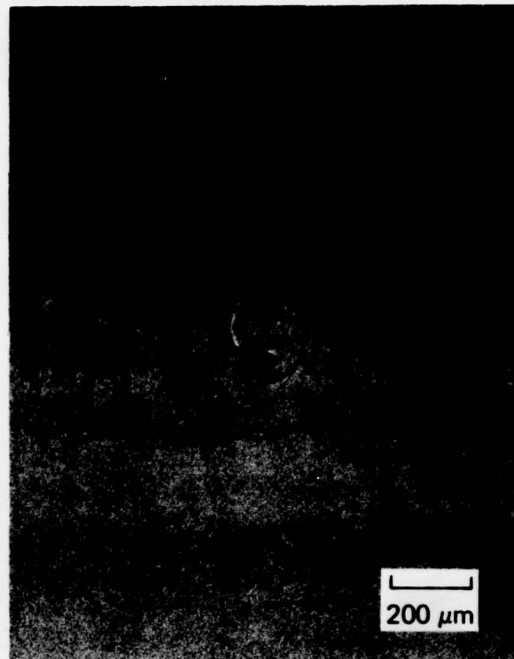
(h) 231 m/s

MP-4928-34

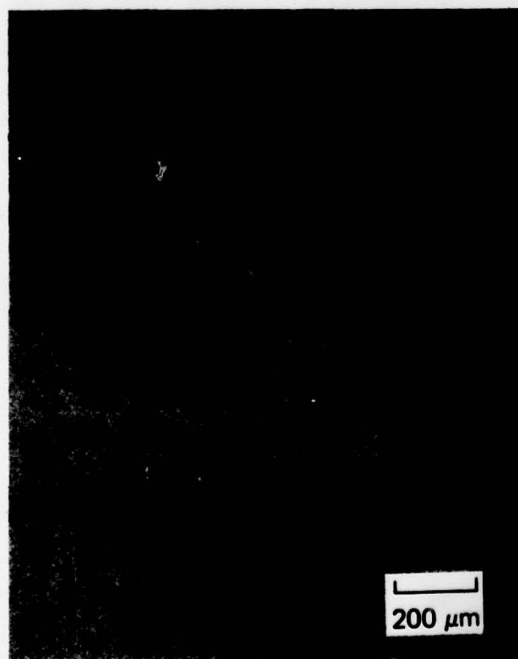
FIGURE 7 TYPICAL IMPACT DAMAGE IN HP Si_3N_4 BY 2.4-mm-DIAMETER WC SPHERES (Impact Configuration B) (Concluded)



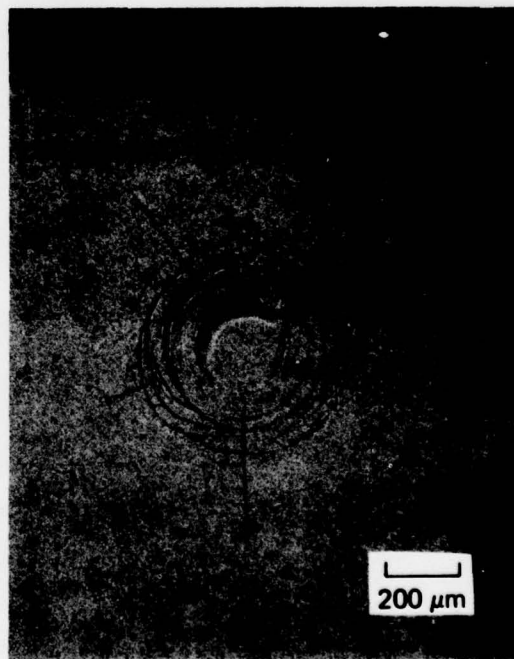
(a) 37.6 m/s



(c) 78.1 m/s



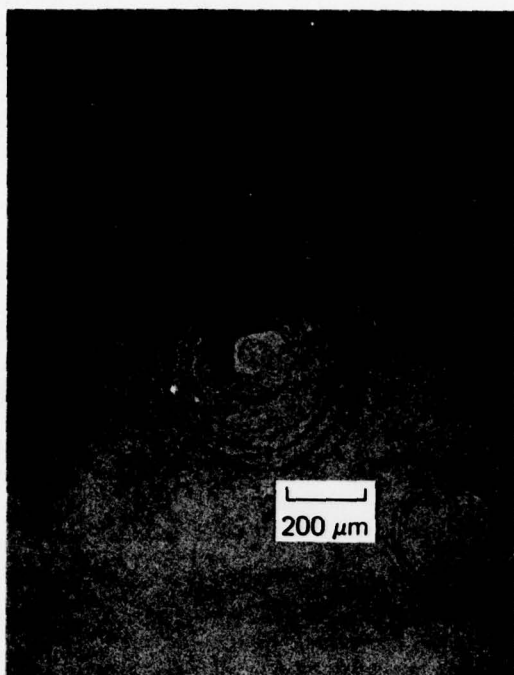
(b) 67.7 m/s



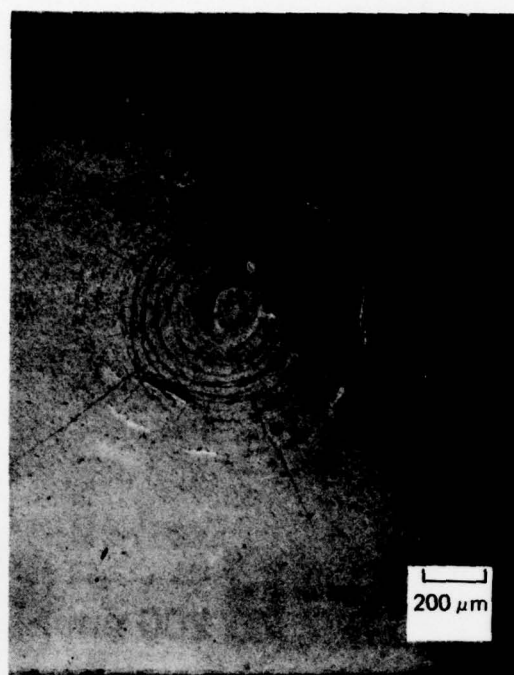
(d) 92.4 m/s

MP-4928-35

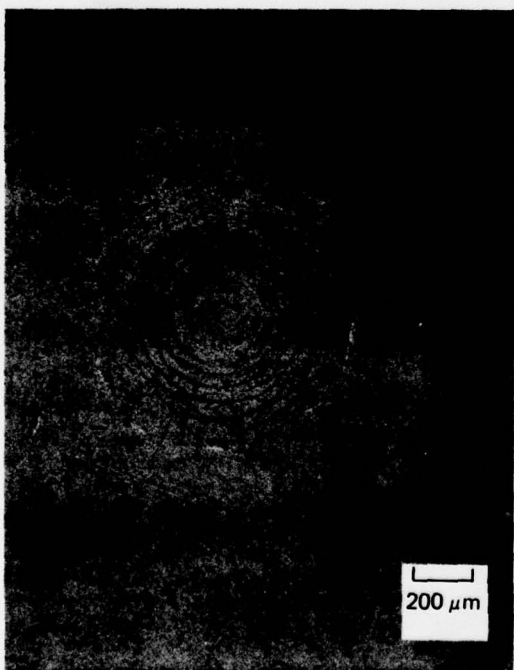
FIGURE 8 TYPICAL IMPACT DAMAGE IN HP Si_3N_4 BY 1.6-mm-DIAMETER WC SPHERES
(Impact Configuration C)



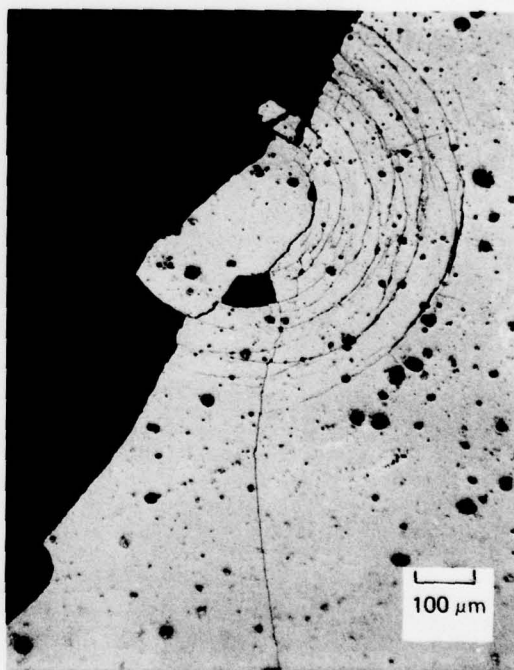
(e) 102 m/s



(g) 195 m/s



(f) 127 m/s



(h) 368 m/s

MP-4928-36

FIGURE 8 TYPICAL IMPACT DAMAGE IN HP Si_3N_4 BY 1.6-mm-DIAMETER WC SPHERES
(Impact Configuration C) (Concluded)

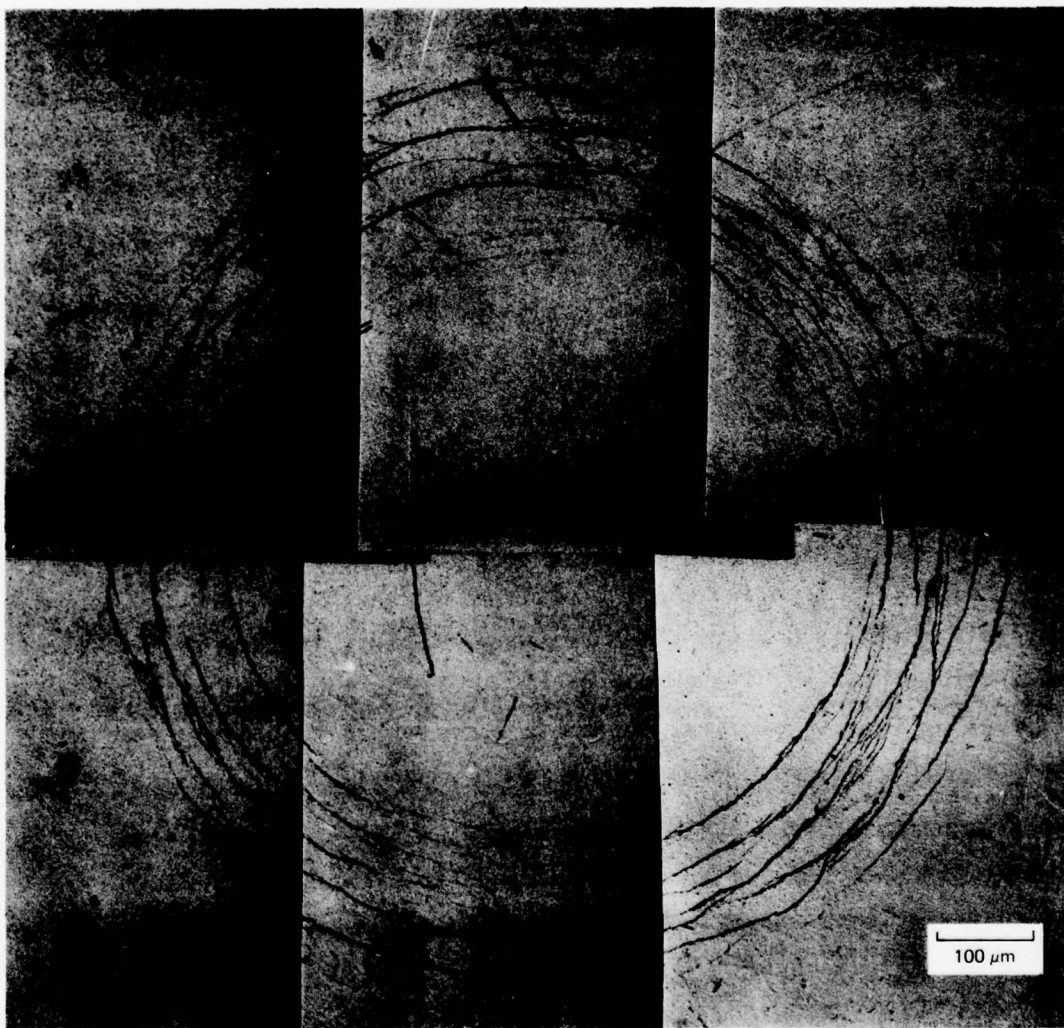
The cracking sequence caused by the 1.6- and 2.4-mm-diameter WC spheres was qualitatively similar. It consisted of ring cracks (shallow circumferential cracks) followed by a plastic impression, radial cracks, lateral cracks, and target fracture.

Damage in HP Si_3N_4 due to 2.4-mm-diameter steel spheres (impact configuration A) consisted exclusively of ring cracks throughout the velocity range. At 299 m/s the steel projectile disintegrated, and no significant increase in target damage was observed beyond this velocity. The white areas in Figure 7(f) seem to result from outflow of material from the disintegrating sphere. Ring cracks started to form at fairly low velocities for all three impact configurations (42.3 m/s, Figure 6; 19.5 m/s, Figure 7; and 37.6 m/s, Figure 8). As impact velocity increased, more and longer ring cracks formed.

The ring cracks appeared to be similar to the Hertzian ring cracks formed under quasi-static loading (Reference 4, for example). A magnified view of a typical population of ring cracks is presented in Figure 9. Most ring cracks were short segmental cracks that seemed to nucleate at an arbitrary location where a particular flaw might exist, propagated circumferentially a short distance, and stopped. There were also some full-circle cracks. The number of these cracks increased strongly with increasing impact velocity, spreading both toward the contact center and to outer regions.

We produced ring cracks by both quasi-static indentation and particle impact and used replica and scanning electron microscopy to examine the influence of microstructure on crack nucleation and growth. The results are described in Section VI.

Slight plastic impressions were first observed at 31.7 and about 42 m/s for impact configurations B and C, respectively. As the velocity increased, the impression became deeper but did not enlarge. Plastic impression and radial cracks seemed to appear at the same time in the cracking sequence. As the impression deepened with increased velocity, the radial cracks grew in both size and number, although only a few (8 or 9) of the radial cracks grew to great lengths (several millimeters).



MP-4928-37

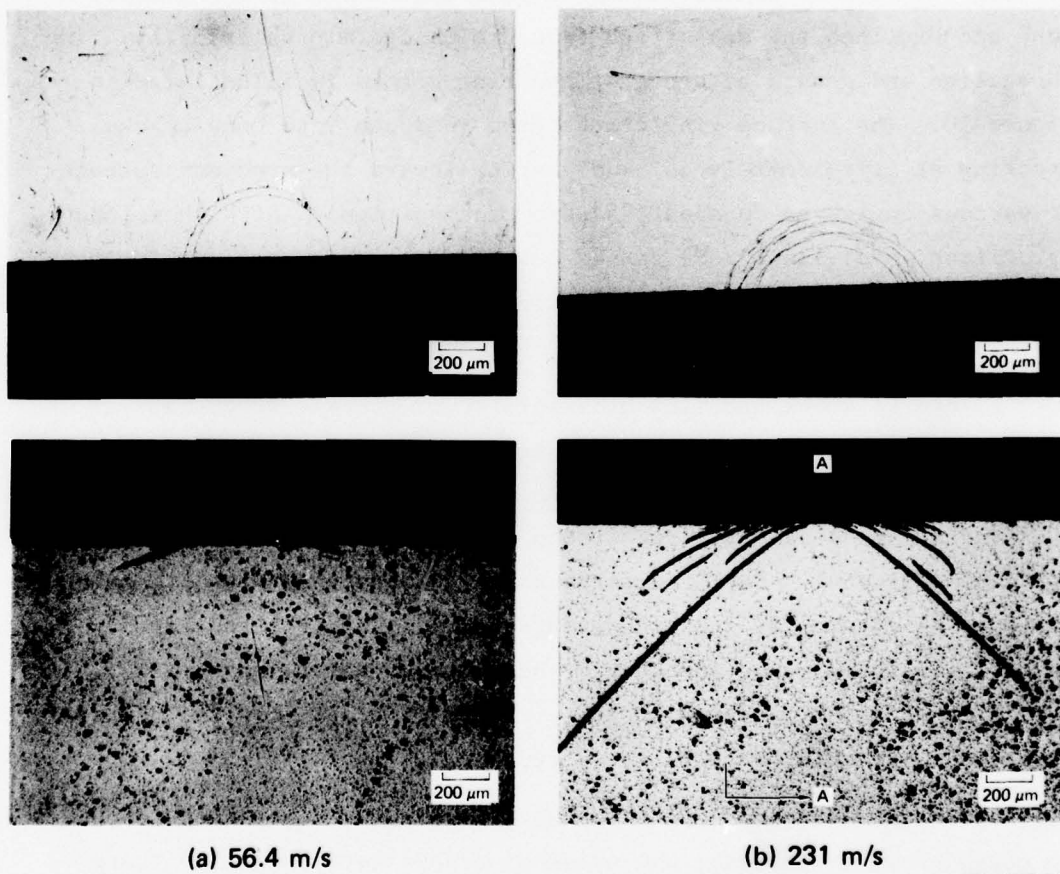
FIGURE 9 A MAGNIFIED VIEW OF TYPICAL RING CRACKS IN HP Si_3N_4 (Particle: 2.4-mm-diameter steel sphere, Velocity: 195 m/s, See also Figure 6d)

Plastic deformation in the target probably modifies the previous elastic stress field outside the contact zone. The hoop stress must become tensile, creating the necessary conditions for radial cracking as observed.

To determine internal damage and the extent of growth of the various cracks below the specimen surface, we sectioned the specimens to expose surfaces at various radial distances from the center of the contact zone and examined the subsurface fracture damage microscopically. The nucleation and growth sequence of the ring cracks is illustrated in Figure 10. The surface ring cracks grew in depth into cone cracks starting at approximately 45° and veering toward the specimen surface at various angles up to about 75° from the vertical. At higher impact velocities, additional cone cracks formed both inside and outside the main cone crack, and the innermost cone grew several millimeters in depth.

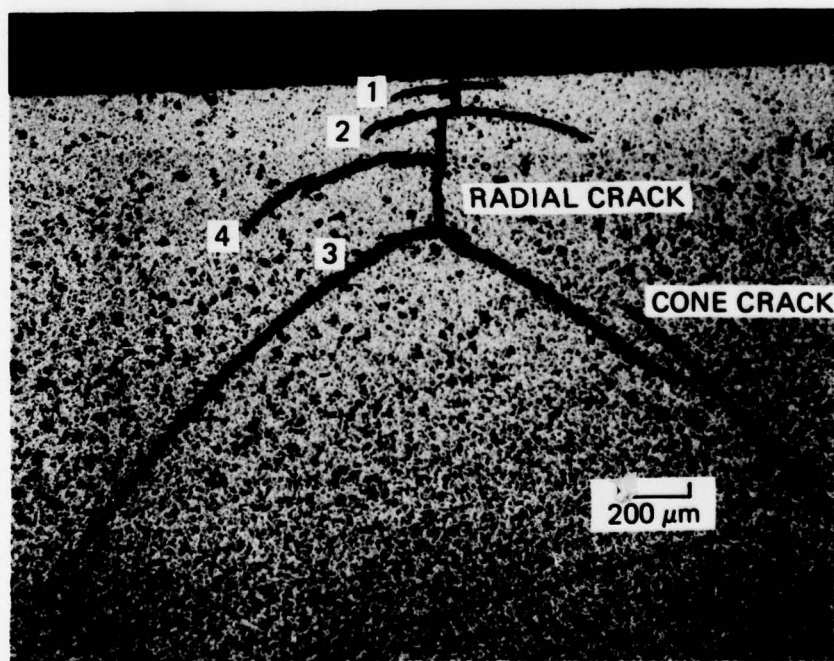
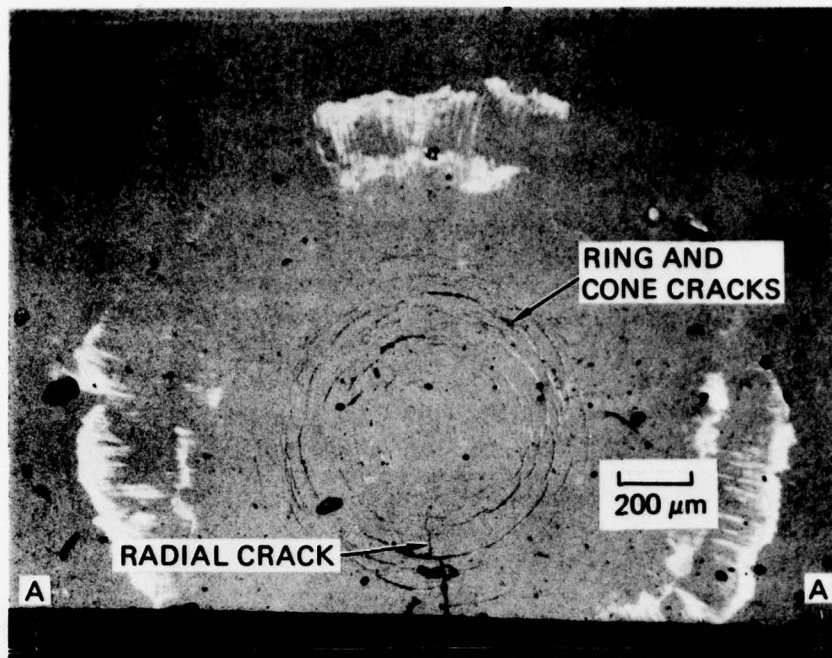
Figure 11 shows profiles of several cone cracks, essentially depicting a section view across the cone cracks of the area marked AA in Figures 10 and 11. Figure 11 also illustrates the surface profile of a radial crack and its interaction with the cone cracks. Cone cracks 1, 2, 3, (Figure 11) appeared first and grew independently of each other followed by the radial crack that intersected the first two cone cracks, reached the third cone, and then branched out. Newer cone cracks seemed to nucleate and grow in the presence of radial cracks, and their growth was often interrupted (cone crack 4 in Figure 11).

Under increasingly severe impacts, Figure 12, cone cracks seemed to cease growing; instead, new types of cracks were created. There were profiles of what appeared to be lateral cracks that nucleated near the contact center, ran approximately parallel to, and eventually veered sharply toward the specimen free surface. There were also vertical cracks that initiated from inside the innermost cone and converged to the damage center as successive sections were examined. We speculate that these are penny-shaped cracks similar to the median-vent cracks observed by Evans and Wilshaw in their quasi-static indentation experiments.⁵ A zone of damage-induced porous material, approximately spherical



MP 4928-38

FIGURE 10 SECTIONAL VIEWS SHOWING SUBSURFACE GROWTH OF RING CRACKS INTO CONE CRACKS IN HP Si_3N_4 (Particles: 2.4-mm-Diameter Steel Spheres)

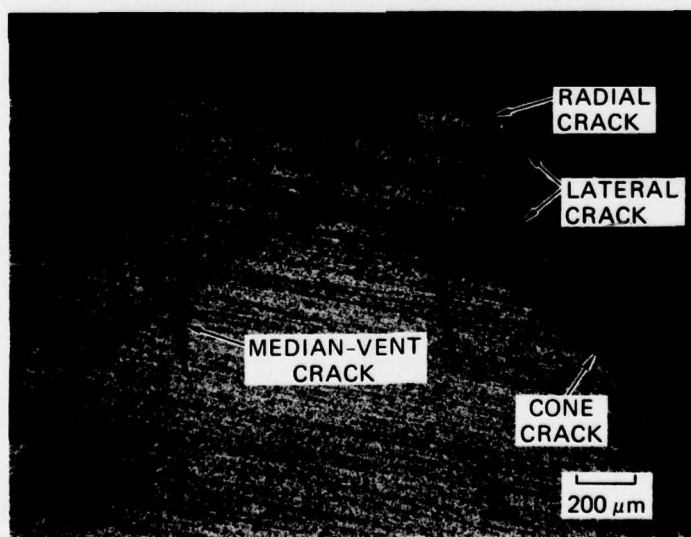
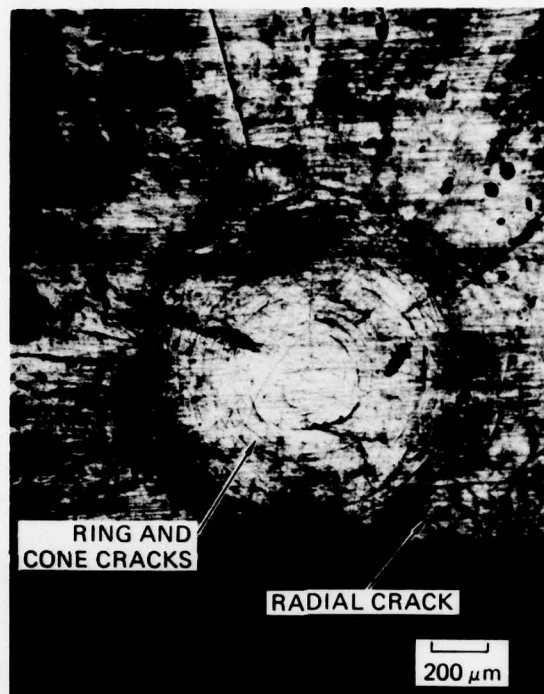


299 m/s

MP-4928-39

FIGURE 11 SECTIONAL VIEW OF SEVERAL CONE CRACKS AND ONE RADIAL CRACK 0.50 mm FROM CONTACT CENTER

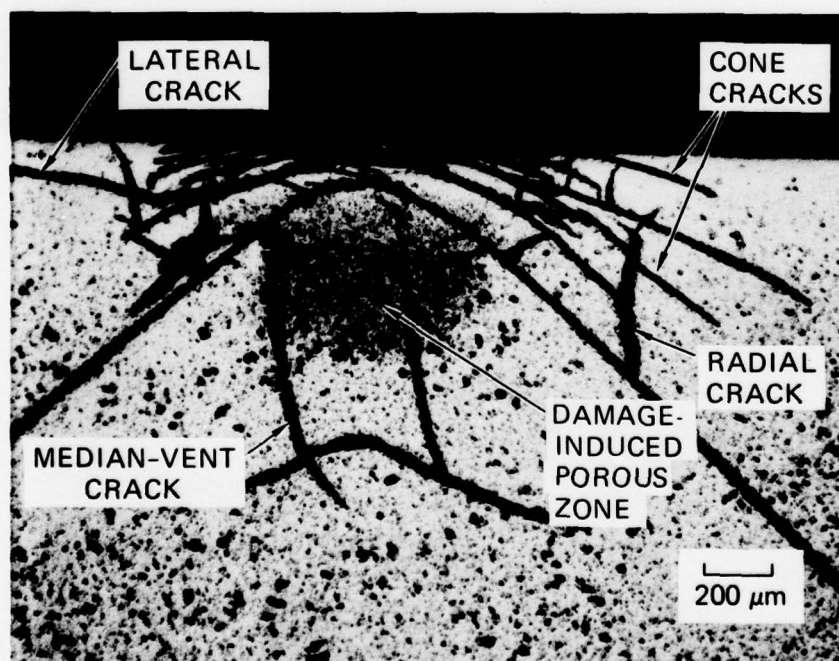
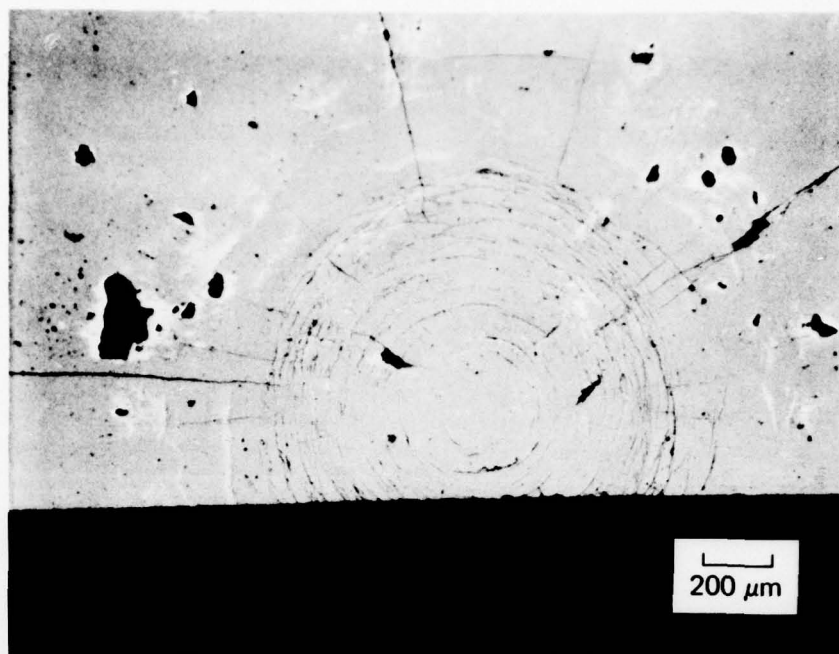
Note the interaction between cone and radial cracks. Particle: steel, 2.4 mm; Target: HP Si_3N_4



(a) 0.60 mm FROM CONTACT CENTER

MP-4928-40

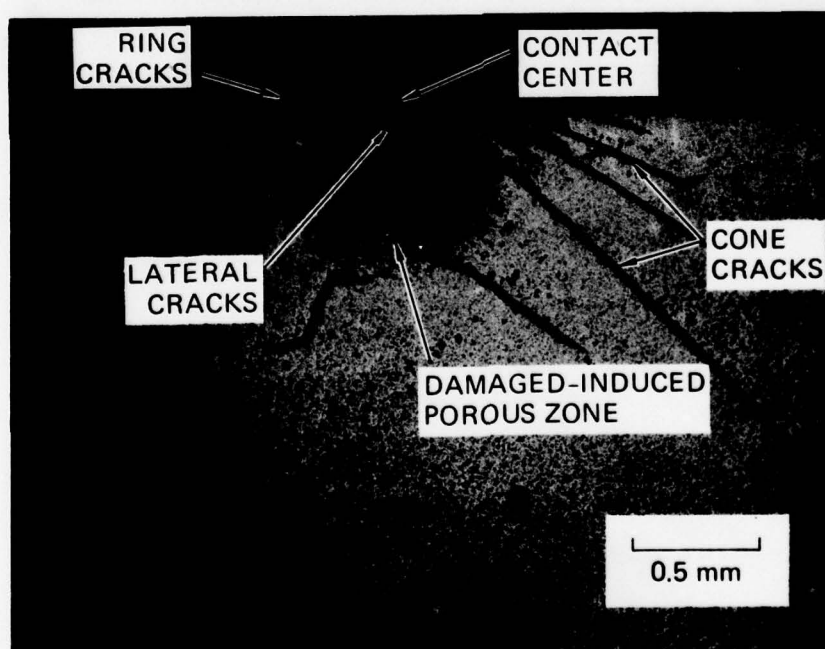
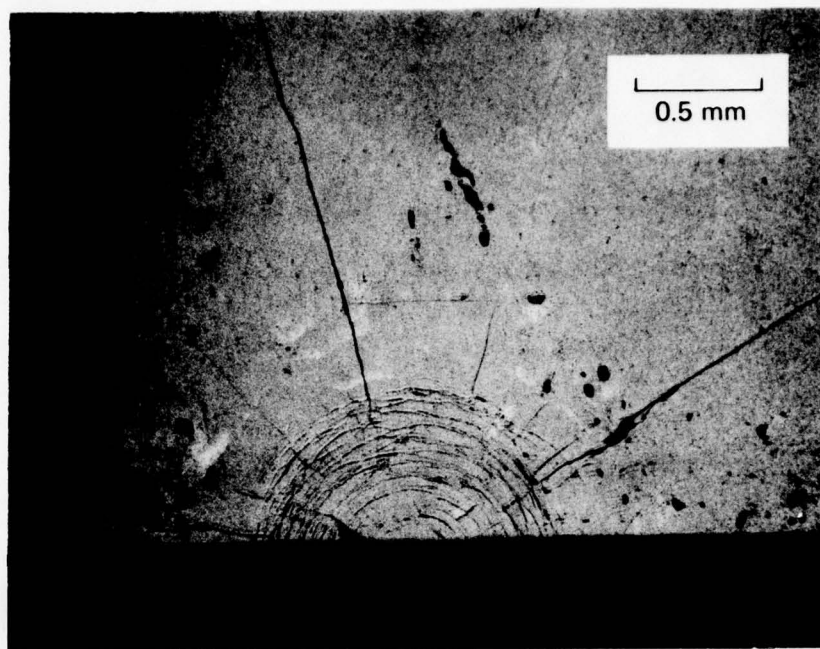
FIGURE 12 SECTIONAL VIEW OF SEVERAL TYPES OF CRACKS IN HP Si_3N_4 IMPACTED AT 231 m/s BY A 2.4-mm-DIAMETER WC SPHERE



(b) 0.25 mm FROM CONTACT CENTER

MP-4928-41

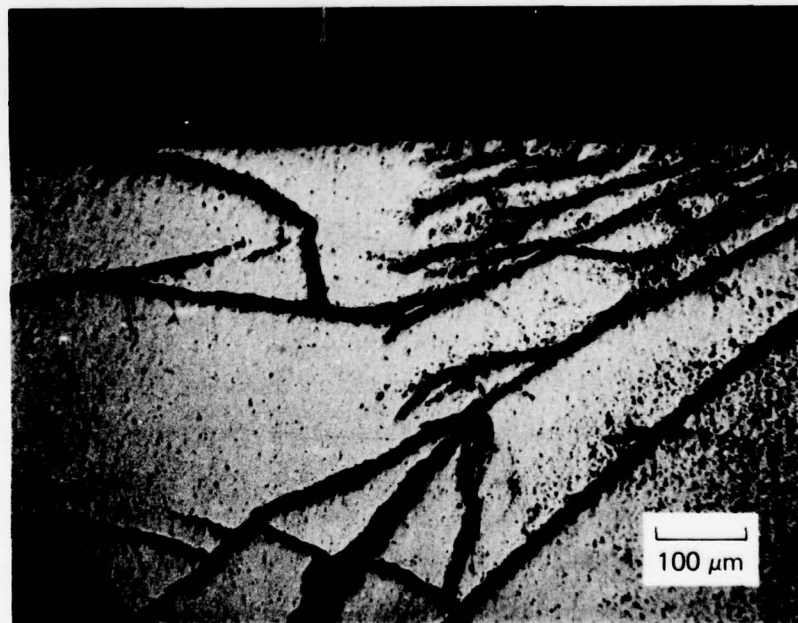
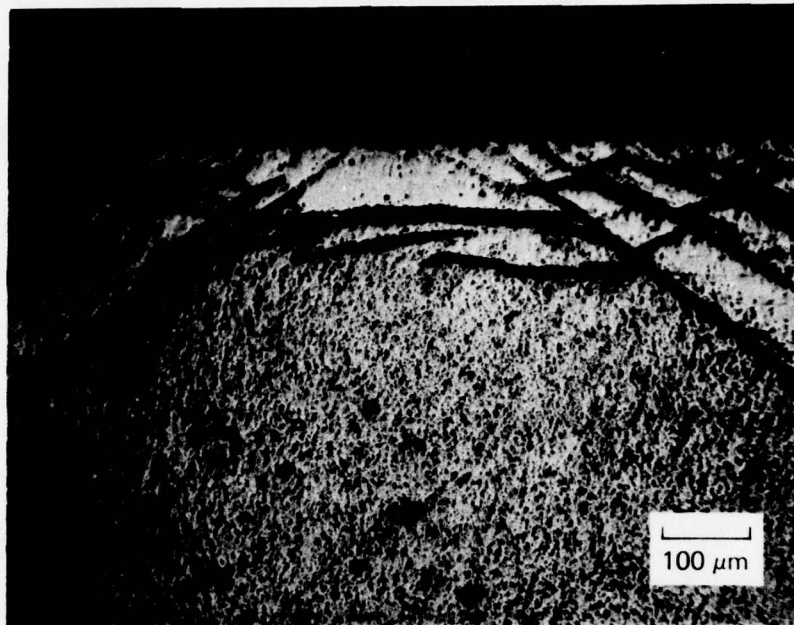
FIGURE 12 SECTIONAL VIEW OF SEVERAL TYPES OF CRACKS IN HP Si_3N_4 IMPACTED AT 231 m/s BY A 2.4-mm-DIAMETER WC SPHERE (Continued)



(c) AT CONTACT CENTER

MP-4928-42

FIGURE 12 SECTIONAL VIEW OF SEVERAL TYPES OF CRACKS IN HP Si_3N_4 IMPACTED AT 231 m/s BY A 2.4-mm-DIAMETER WC SPHERE (Continued)



(d) MAGNIFIED VIEWS OF DAMAGE-INDUCED POROUS
ZONE BENEATH CONTACT CENTER

MP-4928-74

FIGURE 12 SECTIONAL VIEW OF SEVERAL TYPES OF CRACKS IN HP Si_3N_4 IMPACTED
AT 231 m/s BY A 2.4-mm-DIAMETER WC SPHERE (Concluded)

in shape, was also observed beneath the contact area, Figure 12(b) and (c). High-magnification photographs give detail of this porous zone in Figure 12(d). Zinc sulfide impacted by 0.4- and 0.8-mm-diameter WC spheres exhibited a similar porous zone.^{1,6} The failure mechanism responsible for this porous zone is not clearly understood.

Figures 6 and 7 compare impact damage at 231 m/s by spheres of steel and WC. The surface and subsurface damage by the WC sphere is significantly greater.

The foregoing preliminary fractographic observations provide several qualitative conclusions:

- While larger particles produced larger damage zones and cracks, the fracture-damage morphology produced by the 2.4-mm-diameter and 1.6-mm-diameter WC spheres was qualitatively similar.
- The cracking sequence produced by the WC spheres consisted of surface ring cracks with associated cone cracks followed by plastic deformation and radial cracks, lateral cracks, and median-vent cracks. There were strong interactions between these different types of cracks.
- For a similar range of velocity, the softer steel spheres produced only ring cracks, some of which grew into cone cracks. Complete fragmentation of the steel sphere at 299 m/s and little increase in target damage above 299 m/s indicated the inability of the steel spheres to produce other types of damage.

Reaction Bonded Silicon Nitride

Summary of Particle Impact Experiments

We performed 18 particle impact experiments on RB Si_3N_4 with particles of two sizes (1.6 and 0.4 mm in diameter), one particle/target combination (WC sphere/RB Si_3N_4 target), and a range of impact velocities (11.3 to 700 m/s). The experiments were conducted to obtain a wide range of fracture damage in RB Si_3N_4 to investigate the influences of impact velocity and particle size on the damage. The results could also be compared with those obtained for HP Si_3N_4 under similar impact conditions.

Fourteen impacts were carried out on RB Si_3N_4 using the 1.6-mm-diameter WC spheres ranging in impact velocity from 11.3 to 72.6 m/s.

It was discovered during the experiments that low velocities were sufficient for the 1.6-mm-diameter WC spheres to cause substantial damage in the RB Si_3N_4 . The 2.4-mm-diameter WC spheres were not used for similar experiments because it was expected that even lower velocities would be adequate to cause severe damage and the information would be less useful. We decided to conduct several experiments in which sub-millimeter WC spheres were used to raise the impact velocities to a damage level more representative of erosion conditions. Four experiments were performed using a 0.4-mm-diameter WC sphere with impact velocities ranging from 310 to 700 m/s. Table 2 summarizes the important details of these tests.

Fractographic Observations

Typical scanning electron micrographs of the RB Si_3N_4 specimens are presented in Figures 13 through 15. The SEM gave a better resolution to the various damage features than the optical microscope because the large population of dark pores did not reflect light very well when an optical microscope was used.

The damage characteristics found for the RB material were similar to those for the HP material described earlier, although much lower velocities were necessary to cause equivalent damage in the RB material. Observable damage features included ring and cone cracks followed, as impact velocity increased, by a plastic impression; radial, lateral, and median-vent cracks; material removal; and target failure. Plastic impressions were deeper for impacts with the 0.4-mm-diameter spheres (Figure 14). Ring cracks were visible at the bottom of the craters. These ring cracks formed earlier in the impact process, but their surface profile later became closed or healed by the increased compressive stress field and significant plastic flow in the target.

Test 17, Figure 14(c) shows surface interaction of the lateral cracks. They are approximately planar cracks that ran almost parallel to and eventually intersected with the specimen free surface, causing portions of the material between adjacent radial cracks to be removed from the impact site. Test 18, Figure 14(d), performed at about 700 m/s,

Table 2

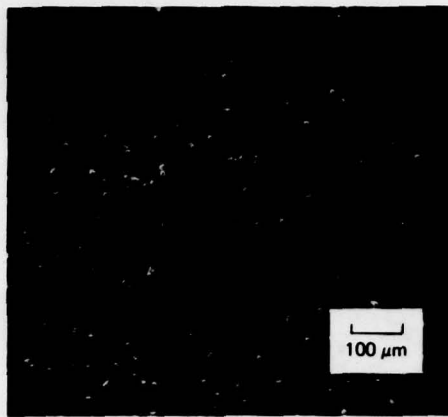
PARTICLE IMPACT EXPERIMENTS ON REACTION BONDED SILICON NITRIDE

Test No.	SRI No.	Impact Velocity (m/s)	Rebound Velocity (m/s)	Damage to Particle
Impact configuration D (1.60-mm-diameter steel, RB Si_3N_4)				
1	4928-76	11.3	11.5 ^a	Intact
2	4928-75	12.7	12.6	b
3	4928-74	15.9	15.4	Intact
4	4928-70	16.9	16.8	Intact
5	4928-73	18.1	20	Intact
6	4928-72	24.2	27	b
7	4928-78	30	34	Intact
8	4928-71	31.7	33	Intact
9	4928-83	46.2	44	Intact
10	4928-85	48	46	b
11	4928-82	48.4	43	b
12	4928-81	58.4	50	b
13	4928-80	65.7	56	b
14	4928-79	72.6	58	b
Impact configuration E (0.40-mm-diameter WC, RB Si_3N_4)				
Impact configuration E WC-0.40/RB Si_3N_4				
15	4928-52	310	c	b
16	4928-50	356	c	b
17	4928-49	506	c	b
18	4928-54	700	c	b

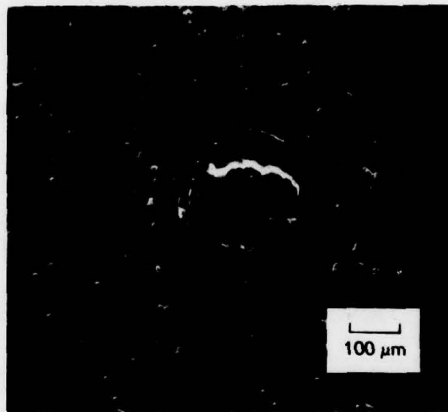
^aHigher rebound velocities indicate increased error in measurements of low velocities (estimated at $\pm 5\%$).

^bParticles were not recovered or no signals from fragments.

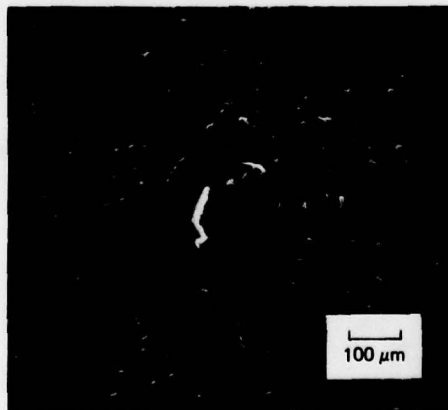
^cRebound velocity not available or particle fragmented.



(a) 11.3 m/s



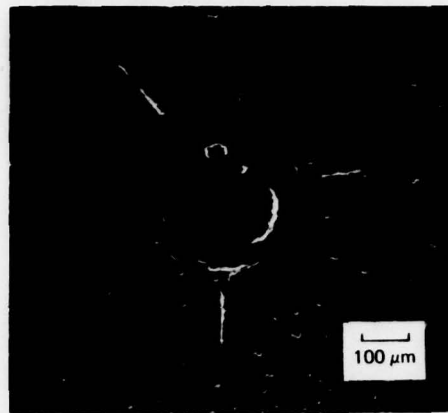
(b) 18.1 m/s



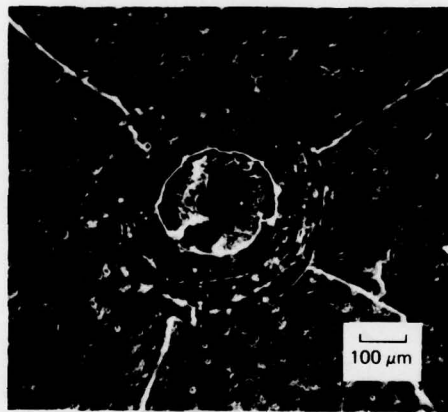
(c) 24.2 m/s

MP-4928-43

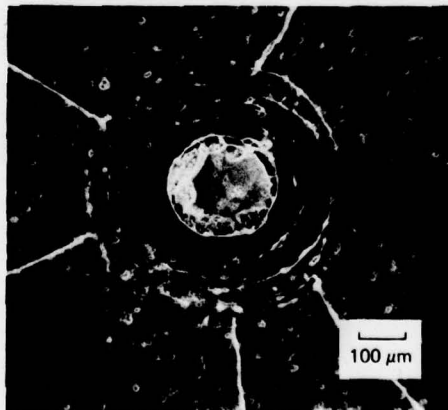
FIGURE 13 TYPICAL IMPACT DAMAGE IN RB Si_3N_4 BY 1.6-mm-DIAMETER WC SPHERES
(Impact Configuration D)



(d) 30 m/s



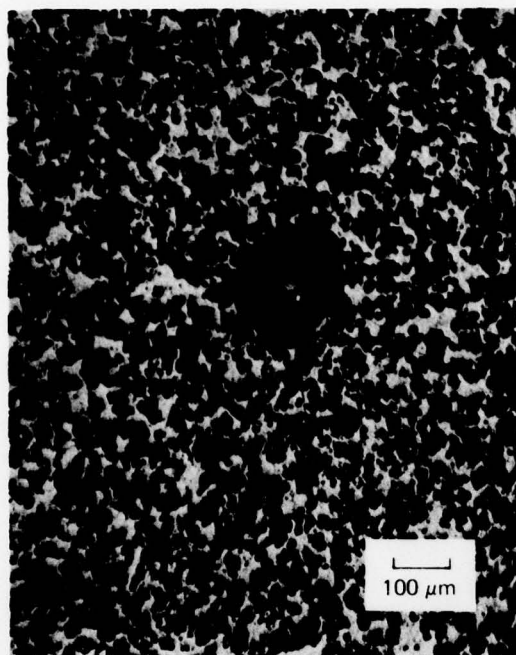
(e) 46.2 m/s



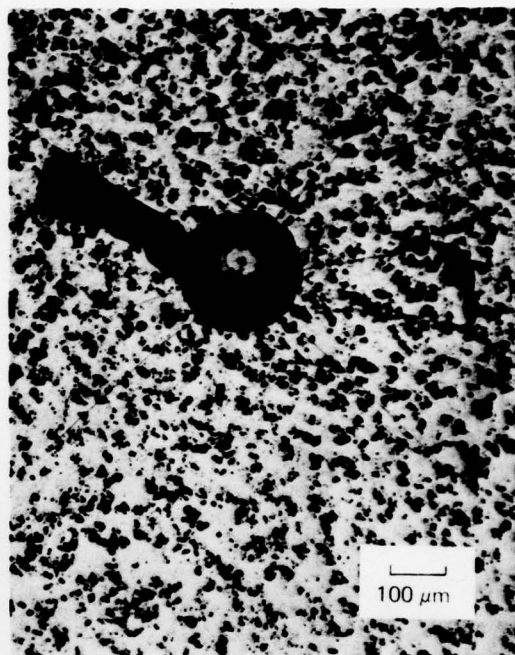
(f) 48.4 m/s

MP-4928-44

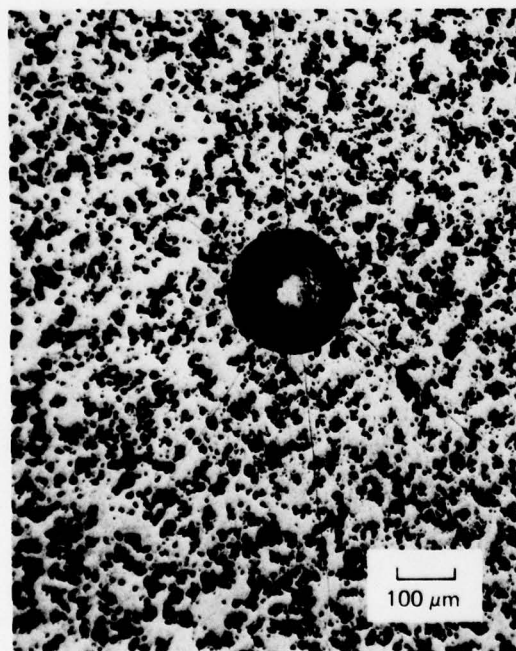
FIGURE 13 TYPICAL IMPACT DAMAGE IN RB Si_3N_4 BY 1.6-mm-DIAMETER WC SPHERES
(Impact Configuration D) (Concluded)



(a) 310 m/s



(c) 506 m/s



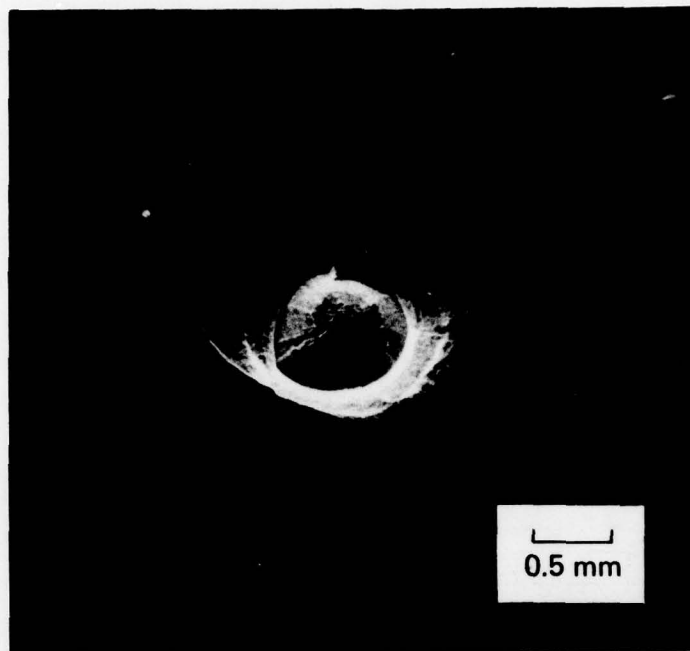
(b) 356 m/s



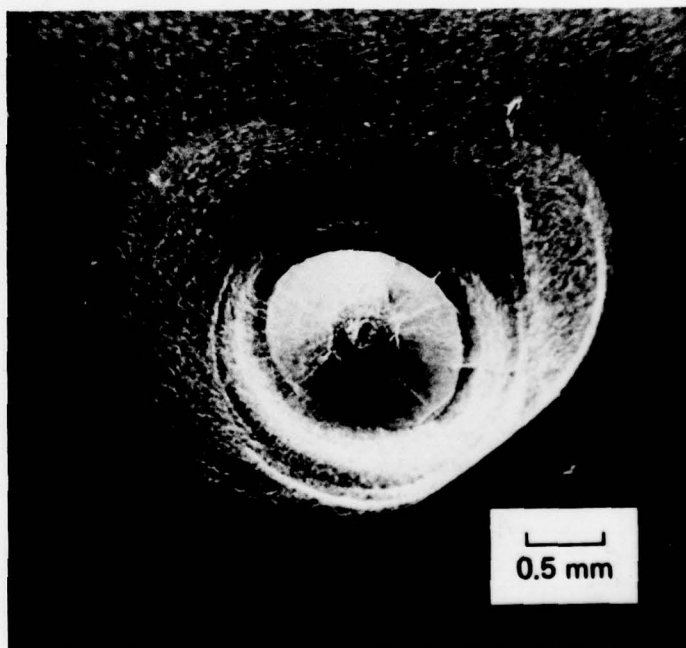
(d) 700 m/s

MP-4928-45

FIGURE 14 TYPICAL IMPACT DAMAGE IN RB Si_3N_4 BY 0.4-mm-DIAMETER WC SPHERES (Impact Configuration E)



(a) 58.4 m/s



(b) 65.7 m/s

MP-4928-46

FIGURE 15 SEVERE SURFACE DAMAGE IN RB Si_3N_4 IMPACTED BY 1.6-mm-DIAMETER WC SPHERES AT VELOCITIES SLIGHTLY HIGHER THAN THOSE IN FIGURE 13

shows increased material removal and reveals the subsurface profile of several lateral cracks. Test 12 and 13 (Figure 15) indicate a more severe case of surface damage in RB Si_3N_4 by larger-size particles (1.6-mm in diameter) when impact velocities were increased only slightly (58-65 m/s) from impacts shown in Figure 13. Profiles of both cone and lateral cracks are visible in the craters. Interactions of these two types of cracks produced surface fracture and material removal.

Oxidized Hot Pressed Silicon Nitride

Summary of Particle Impact Experiments

We performed 16 experiments in which HP Si_3N_4 specimens covered with a layer of oxide scale approximately 20 μm thick were impacted with 2.4-mm-diameter WC spheres (impact configuration F) at 13.4 to 196 m/s. The experiments were performed to investigate the fracture behavior of the oxide scale and the effect of the oxide scale on the fracture response of the underlying HP Si_3N_4 . Table 3 lists these experiments.

Fractographic Observations

Typical scanning electron micrographs of the oxidized specimens impacted at various velocities by the 2.4-mm-diameter WC spheres are shown in Figure 16. The unpolished rugged and porous oxide scale did not reflect light very well. The SEM was therefore used to better resolve the various damage details. Damage features observed on the oxide scale included local spallation, ring cracks, and scattered radial fractures. The spallations at low velocities (13.4-50.8 m/s) were rather small. As the impact velocity was raised, the spalled area broadened and ring cracks formed in the crater. Radial fractures were observed in tests at high velocities (> 72.5 m/s), appearing as small spalled areas (arrowed in Figure 16) scattered around the rim of the crater. The mechanism that controlled the formation of these radial fractures may be linked with the same mechanism that initiated and extended the radial cracks observed in the underlying host material.

Table 3

PARTICLE IMPACT EXPERIMENTS ON OXIDIZED HOT PRESSED SILICON NITRIDE

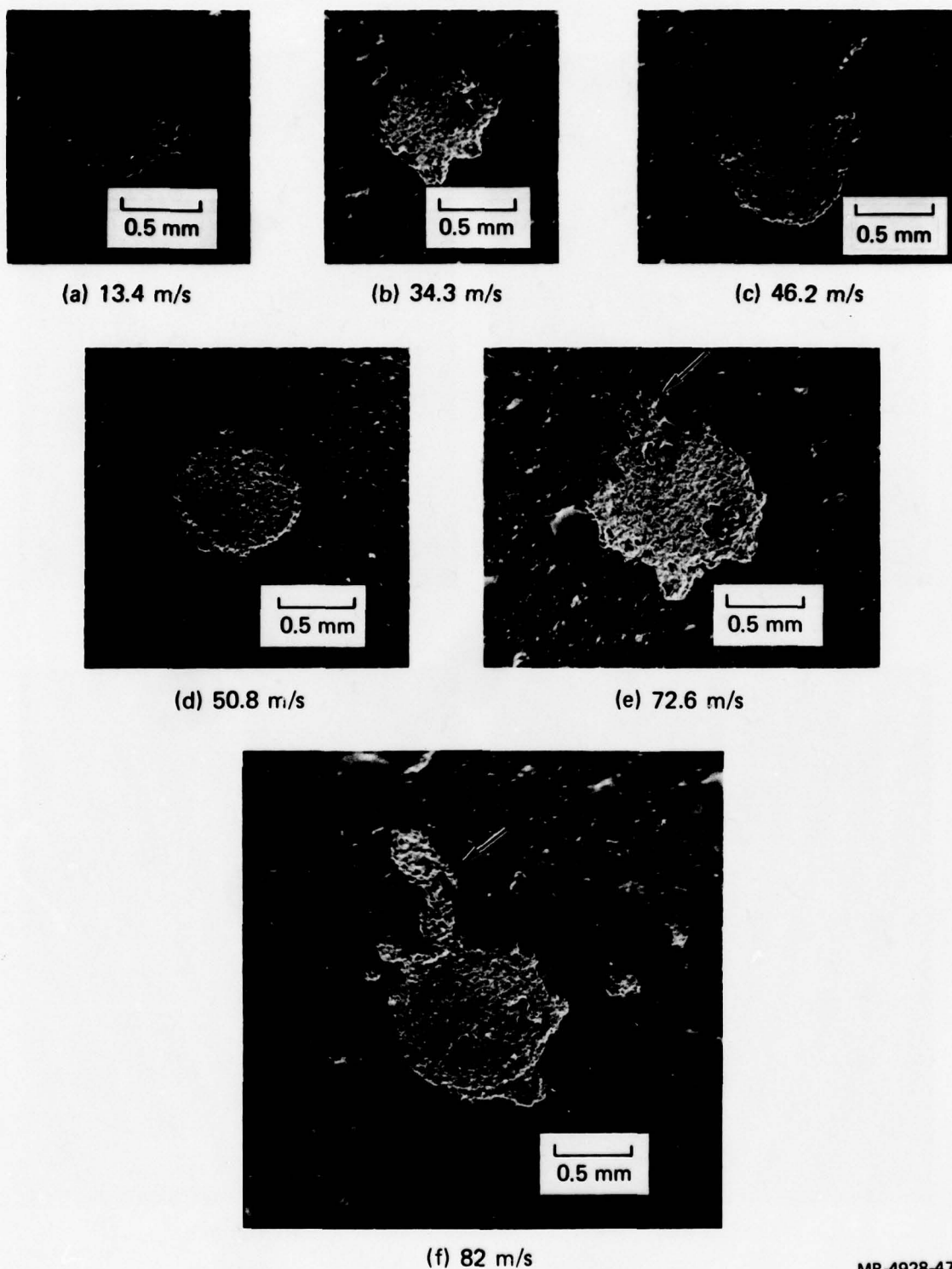
Test No.	SRI No.	Impact Velocity (m/s)	Rebound Velocity (m/s)	Damage to Particle
Impact configuration F (2.40-mm-diameter WC, oxidized HP Si ₃ N ₄)				
1	4928-145	13.4	a	Intact
2	4928-143	14.5	a	b
3	4928-142	16.9	18.5 ^c	Intact
4	4928-144	25.4	24	Intact
5	4928-148	34.3	a	b
6	4928-147	46.2	31	Flattened
7	4928-146	50.8	39	Flattened
8	4928-150	72.6	42	b
9	4928-149	72.6	29	Fragmentation ^d
10	4928-141	82	29	Fragmentation ^d
11	4928-140	84.7	a	b
12	4928-139	113	a	Fragmentation ^d
13	4928-138	145	a	Fragmentation ^d
14	4928-135	~150	a	b
15	4928-136	169	a	Fragmentation ^d
16	4928-137	169	a	Fragmentation ^d

^aRebound velocity not available or particle fragmented.

^bParticles were not recovered or no signals from fragments.

^cHigher rebound velocity indicates increased error in measurement of low velocities (estimated at $\pm 5\%$).

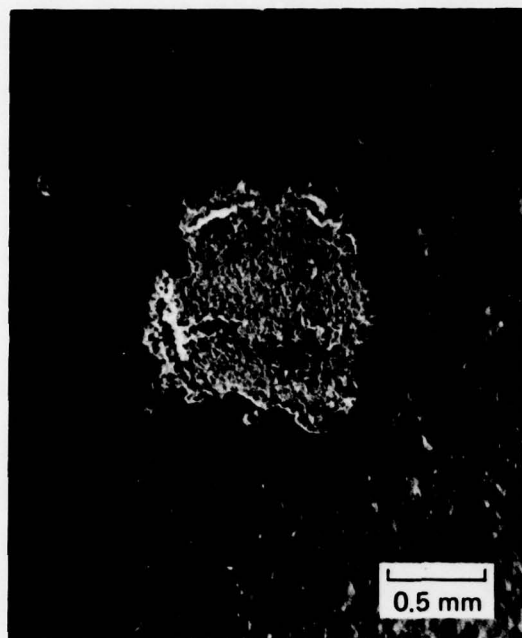
^dDetermined from photomultiplier signals of rebounded fragments.



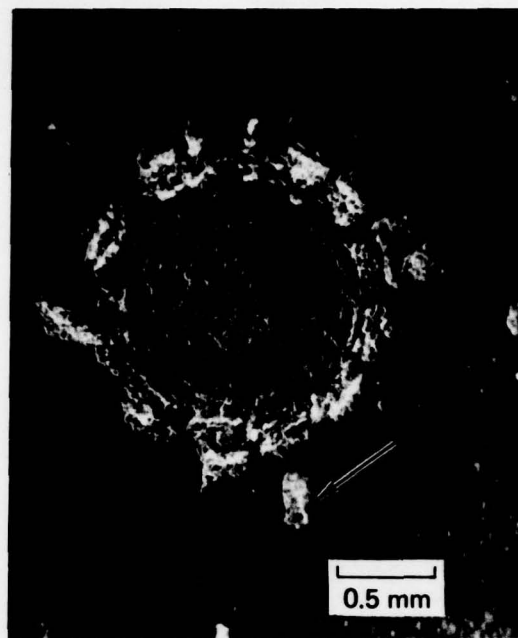
MP-4928-47

FIGURE 16 TYPICAL IMPACT DAMAGE IN OXIDIZED HP Si_3N_4 BY 2.4-mm-DIAMETER WC SPHERES (Impact Configuration F)

Arrows point to radial fractures.



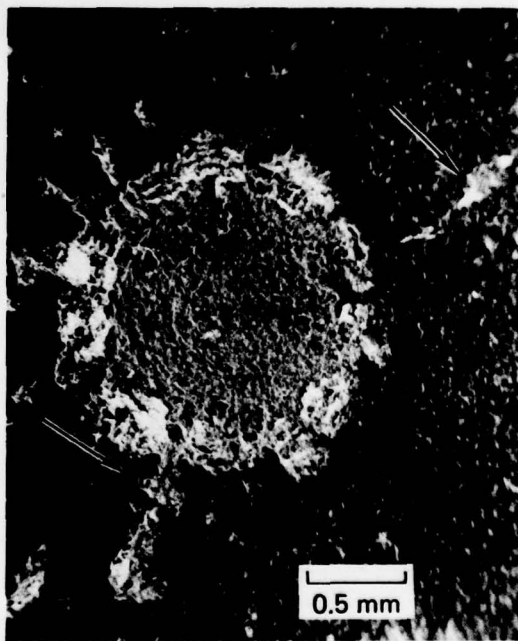
(g) 84.7 m/s



(i) 150 m/s



(h) 113 m/s



(j) 169 m/s

MP4928-48

FIGURE 16 TYPICAL IMPACT DAMAGE IN OXIDIZED HP Si_3N_4 BY 2.4-mm-DIAMETER WC SPHERES (Impact Configuration F)

Arrows point to radial fractures. (Concluded)

To determine internal damage in the oxide layer and in the host material, we removed successive layers of the oxide scale from the surface of the specimens. For test 14 at 150 m/s, the damage observed at different depths is illustrated in Figure 17. In the crater area where the impact spalled the specimen free of oxide material, the damage revealed was that sustained in the host material as shown by all four pictures in Figure 17, where a pattern of ring cracks was observed. Outside the crater zone, as successive layers of the oxide scale were removed by polishing (Figure 17a to c), no cracking was observed in the oxide. This was not surprising because the oxide scale was highly porous.

When the oxide layer was completely removed by polishing, Figure 17d, we observed damage in the substrate that consisted of ring cracks as well as radial cracks. Figure 18 illustrates substrate damage in different tests as a function of impact velocity. Surface damage to the oxide scale and to the HP Si_3N_4 substrate can be compared by comparing Figures 16 and 18.

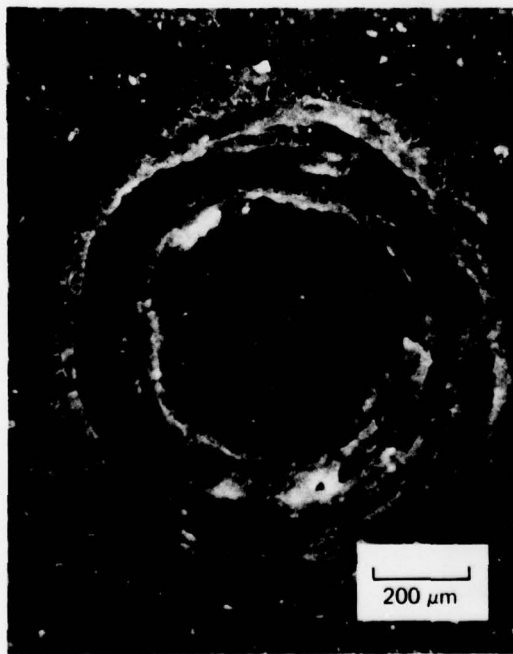
The cracking sequence in the HP Si_3N_4 substrate was similar to that observed for the unoxidized material under similar impact conditions (compare Figures 7 and 18). The crack size distributions are compared quantitatively in Section VII.



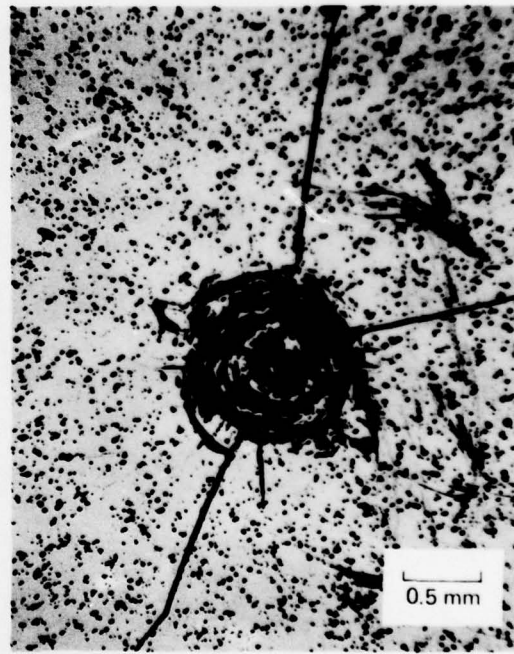
(a) SURFACE



(c) ~ 15 μm BELOW SURFACE



(b) ~ 8 μm BELOW SURFACE

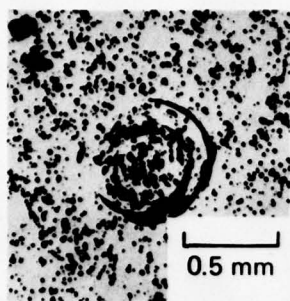


(d) OXIDE SCALE COMPLETELY REMOVED

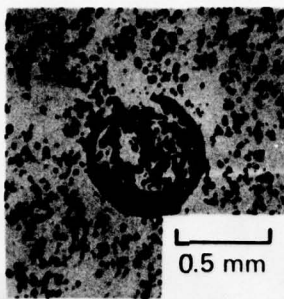
MP-4928-49

FIGURE 17 IMPACT DAMAGE VIEWED AT SUCCESSIVE DEPTHS (Oxidized HP Si_3N_4 Impacted at 150 m/s with a 2.4-mm-Diameter WC Sphere)

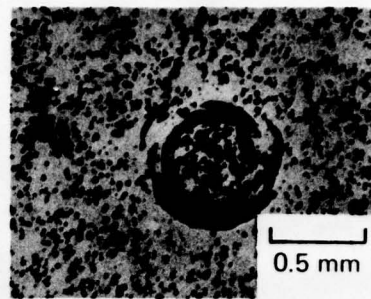
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TO ACCOMMODATE FIGURE 18 BEING ON FACING PAGES.



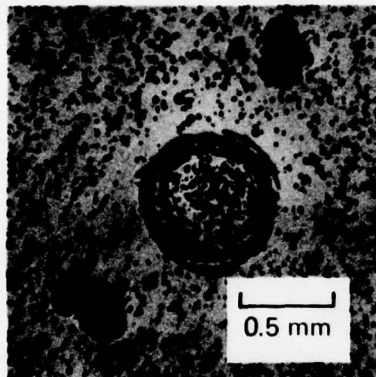
(a) 13.4 m/s



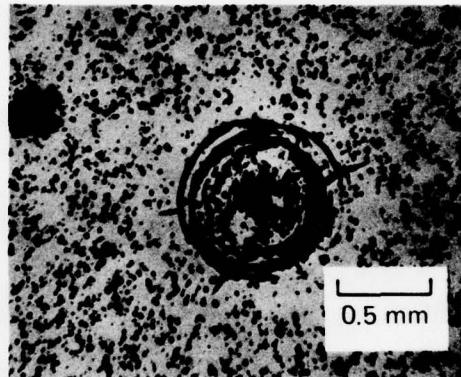
(b) 34.3 m/s



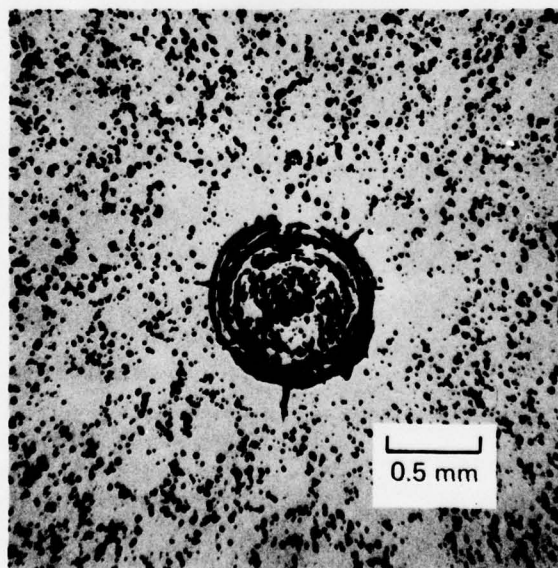
(c) 46.2 m/s



(d) 50.8 m/s



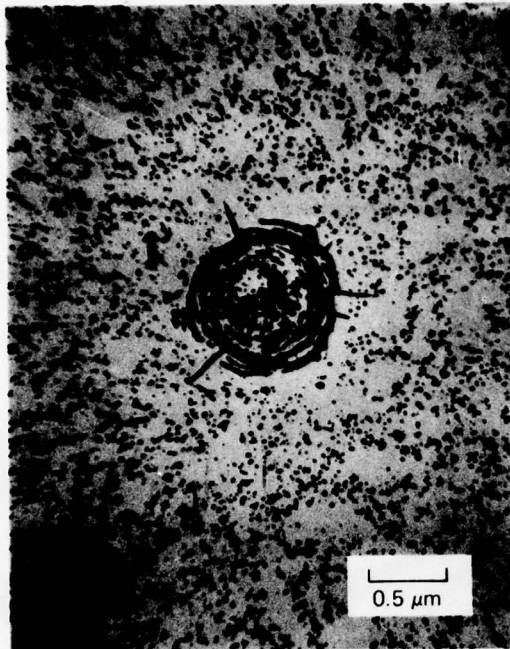
(e) 72.6 m/s



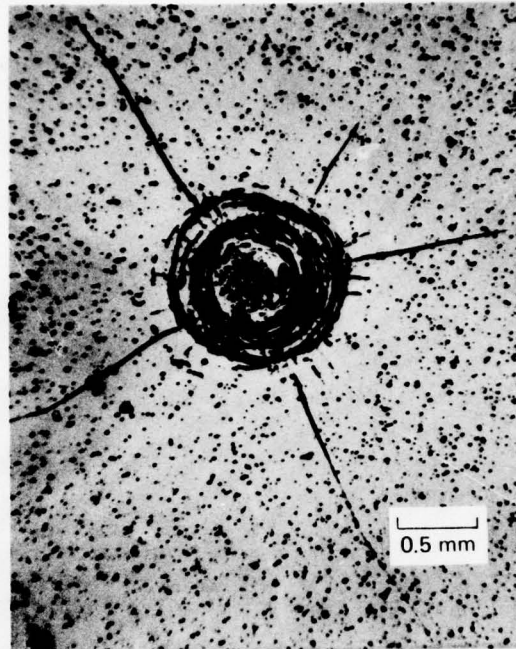
(f) 82 m/s

MP-4928-50

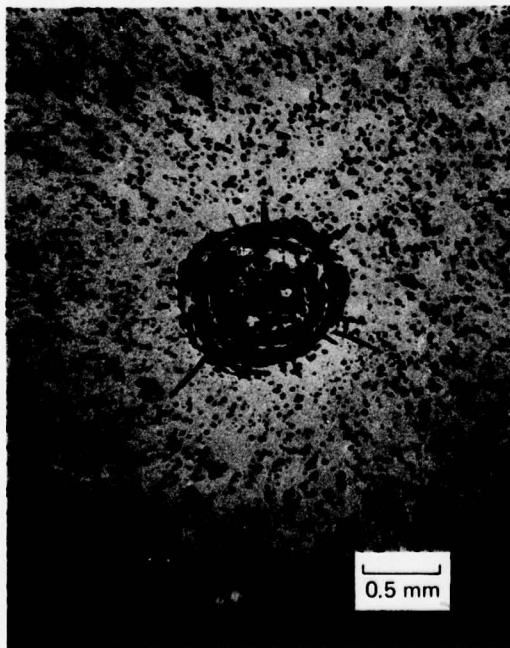
FIGURE 18 TYPICAL IMPACT DAMAGE IN HP Si_3N_4 SUBSTRATE (Beneath the Oxide Scale) BY 2.4-mm-DIAMETER WC SPHERES (Impact Configuration F)



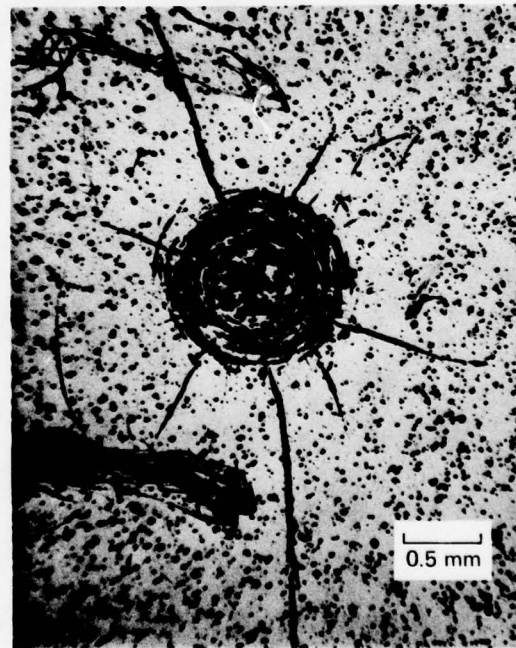
(g) 84.7 m/s



(i) 150 m/s



(h) 113 m/s



(j) 169 m/s

MP-4928-51

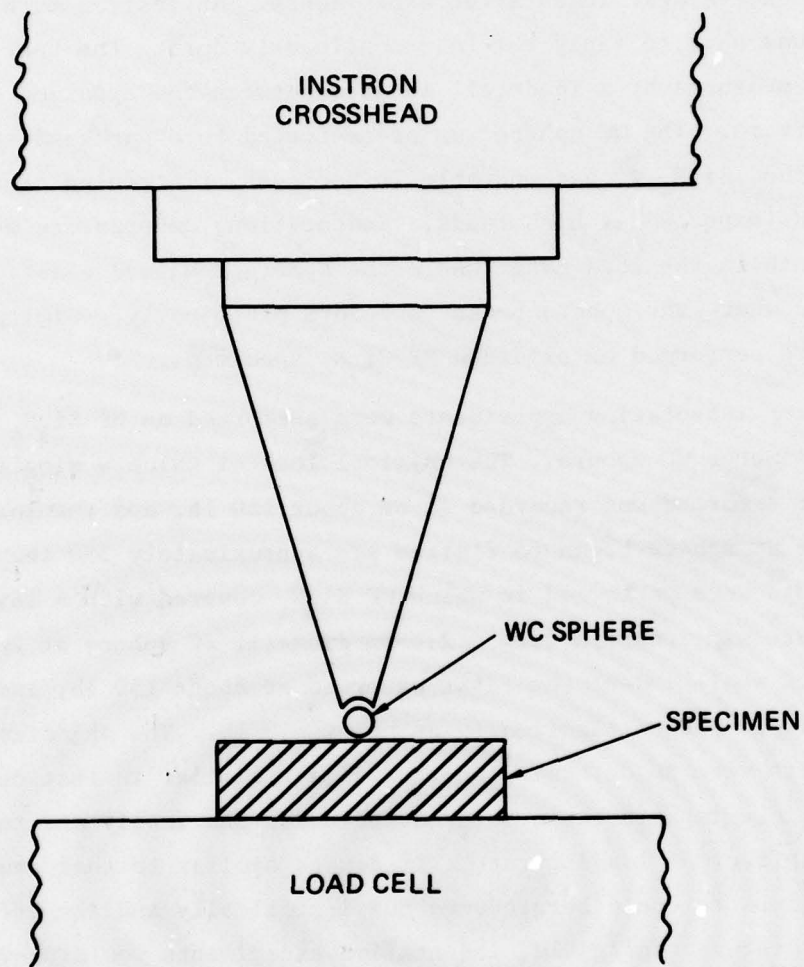
FIGURE 18 TYPICAL IMPACT DAMAGE IN HP Si_3N_4 SUBSTRATE (Beneath the Oxide Scale) BY 2.4-mm-DIAMETER WC SPHERES (Impact Configuration F) (Concluded)

IV QUASI-STATIC INDENTATION EXPERIMENTS

The test fixture illustrated schematically in Figure 19 was used for all quasi-static indentation experiments. An Instron machine, model TT-C-L, was used to apply the load continuously during the test, and the load was measured by a load cell located between the specimen and the machine frame. The WC sphere was press-fitted to a hardened-steel cone. Since WC and Si_3N_4 were comparable in hardness, flattening of the WC sphere was expected at high loads. Indentation experiments were conducted both in the load range where the sphere remained elastic and in the range where the sphere began to deform plastically. Similar experiments were performed on oxidized HP Si_3N_4 specimens.

Eleven indentation experiments were performed on HP Si_3N_4 using a 2.4-mm-diameter WC sphere. The critical load at which a single ring crack first formed was recorded to be about 180 lb, and the load at which the WC sphere began to flatten was approximately 550 lb. Twelve experiments were performed in which HP Si_3N_4 covered with a layer of oxide scale was indented with a 2.4-mm-diameter WC sphere at 200 to 800 lb. A small indent was first observed at about 150 lb, and the ball began to deform plastically at about 550 lb. The objectives of these tests were to determine whether damage similar to that obtained with particle impacts could be produced quasi-statically and to investigate the effect of loading rate. If damage similar to that caused by particle impacts could be produced quasi-statically and the effect of loading rate was negligible, indentation experiments would provide a simpler means to study impact erosion behavior of the material. Table 4 lists all the indentation experiments.

Optical micrographs of surface damage in HP Si_3N_4 specimens indented at various loads by the 2.4-mm-diameter WC spheres are presented in Figure 20. We observed the development of the ring cracks with increasing load. Above 500 lb the indenter began to flatten rapidly



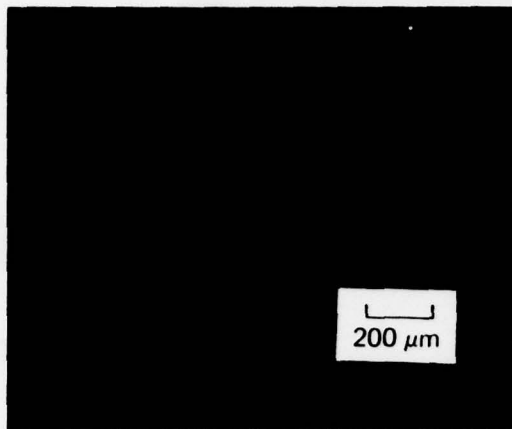
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FIGURE 19 TEST FIXTURE FOR QUASI-STATIC INDENTATION EXPERIMENTS

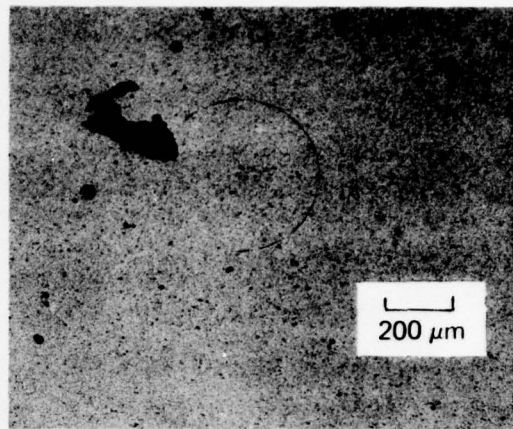
Table 4

QUASI-STATIC INDENTATION EXPERIMENTS

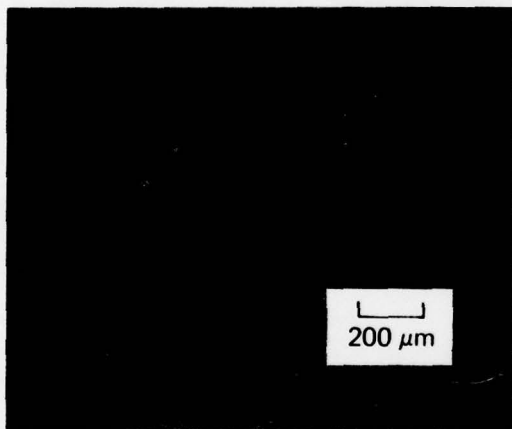
<u>Test No.</u>	<u>SRI No.</u>	<u>Indentation Load (lb)</u>	<u>(N)</u>	<u>Damage to Indenter</u>	<u>Damage to Specimen</u>
2.40-mm-diameter WC, HP Si ₃ N ₄					
1	4928-160	200	(890)	Intact	
2	4928-161	250	(1112)	Intact	
3	4928-162	300	(1334)	Intact	
4	4928-163	350	(1557)	Intact	
5	4928-164	400	(1780)	Intact	
6	4928-165	500	(2224)	Intact	
7	4928-166	500	(2224)	Intact	
8	4928-167	600	(2669)	Flattened	
9	4928-168	700	(3114)	Flattened	
10	4928-169	800	(3558)	Flattened	
11	4928-170	850	(3781)	Flattened	
2.40-mm-diameter WC, Oxidized HP Si ₃ N ₄					
12	4928-171	50	(222)	Intact	
13	4928-172	100	(444)	Intact	
14	4928-173	150	(667)	Intact	
15	4928-174	200	(890)	Intact	
16	4928-175	250	(1112)	Intact	
17	4928-176	300	(1334)	Intact	
18	4928-177	350	(1557)	Intact	
19	4928-178	400	(1780)	Intact	
20	4928-179	500	(2224)	Intact	
21	4928-180	600	(2669)	Flattened	
22	4929-181	700	(3114)	Flattened	
23	4928-182	800	(3558)	Flattened	



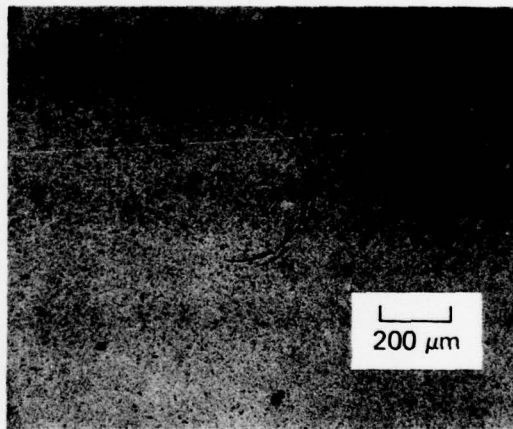
(a) 200 lb (890 N)



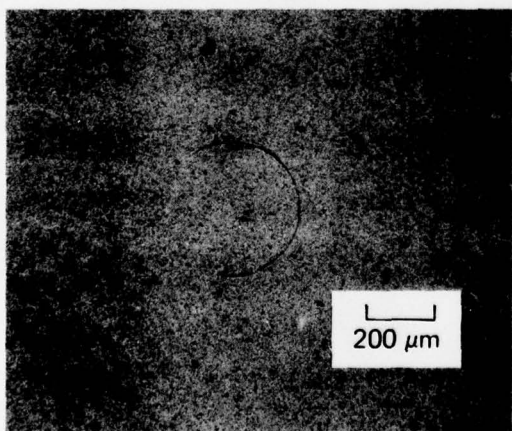
(d) 350 lb (1557 N)



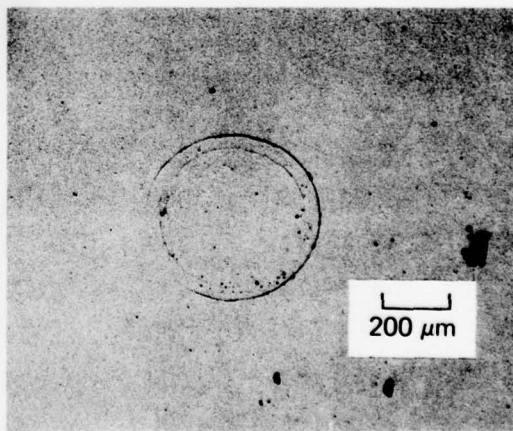
(b) 250 lb (1112 N)



(e) 400 lb (1780 N)



(c) 300 lb (1334 N)



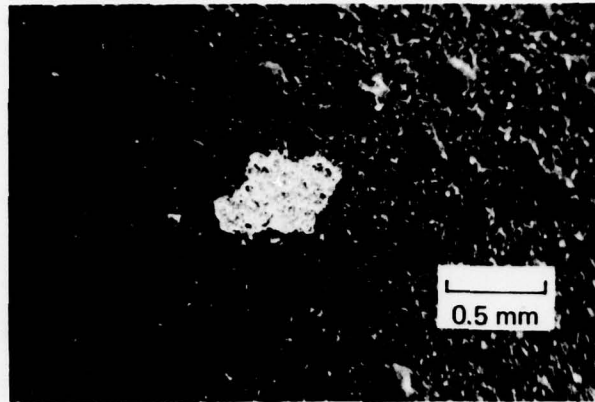
(f) 500 lb (2224 N)

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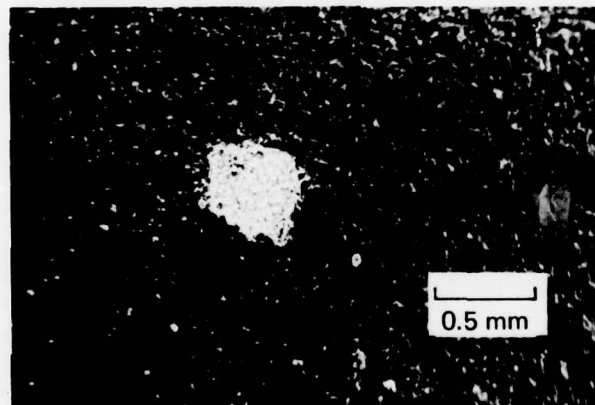
FIGURE 20 TYPICAL QUASI-STATIC-INDENTATION DAMAGE IN HP Si_3N_4 BY 2.4-mm-DIAMETER WC SPHERES

with load, and no further increase in specimen damage was observed. The results indicate that quasi-static particle contact produced incipient ring cracks but could not induce second-stage damage such as plastic impression and radial, lateral, and median-vent cracks similar to that sustained under impact conditions. At high loads the indenter deformed plastically and did not transfer the increase in load or pressure necessary for additional specimen damage. In particle impact experiments sufficiently high loads could be applied to the specimens by the impacting spheres for short durations.

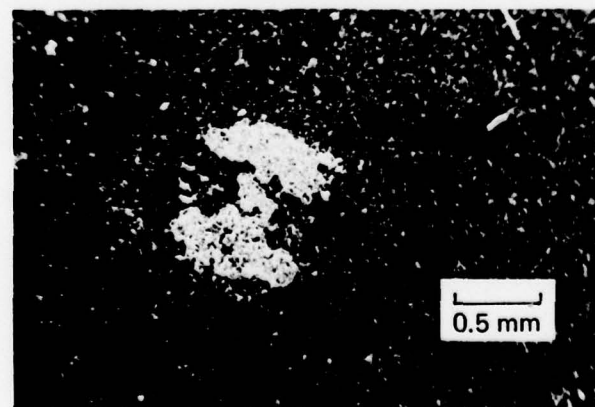
Scanning electron micrographs of the oxide scale indented at three representative loads by the 2.4-mm-diameter WC sphere are shown in Figure 21. The scale broke off in a layer similar to local spallations by particle impacts at low velocities (Figure 16). There were no observable surface ring cracking or radial fractures produced quasi-statically. We conclude from these observations, without further analysis, that oxidized and unoxidized HP Si_3N_4 behaved similarly under quasi-static indentation, and that high levels of impact erosion damage can not be easily duplicated quasi-statically for silicon nitride because plastic deformation of the indenter prevents sufficient loads from being applied.



(a) 300 lb (1334 N)



(b) 400 lb (1780 N)



(c) 500 lb (2224 N)

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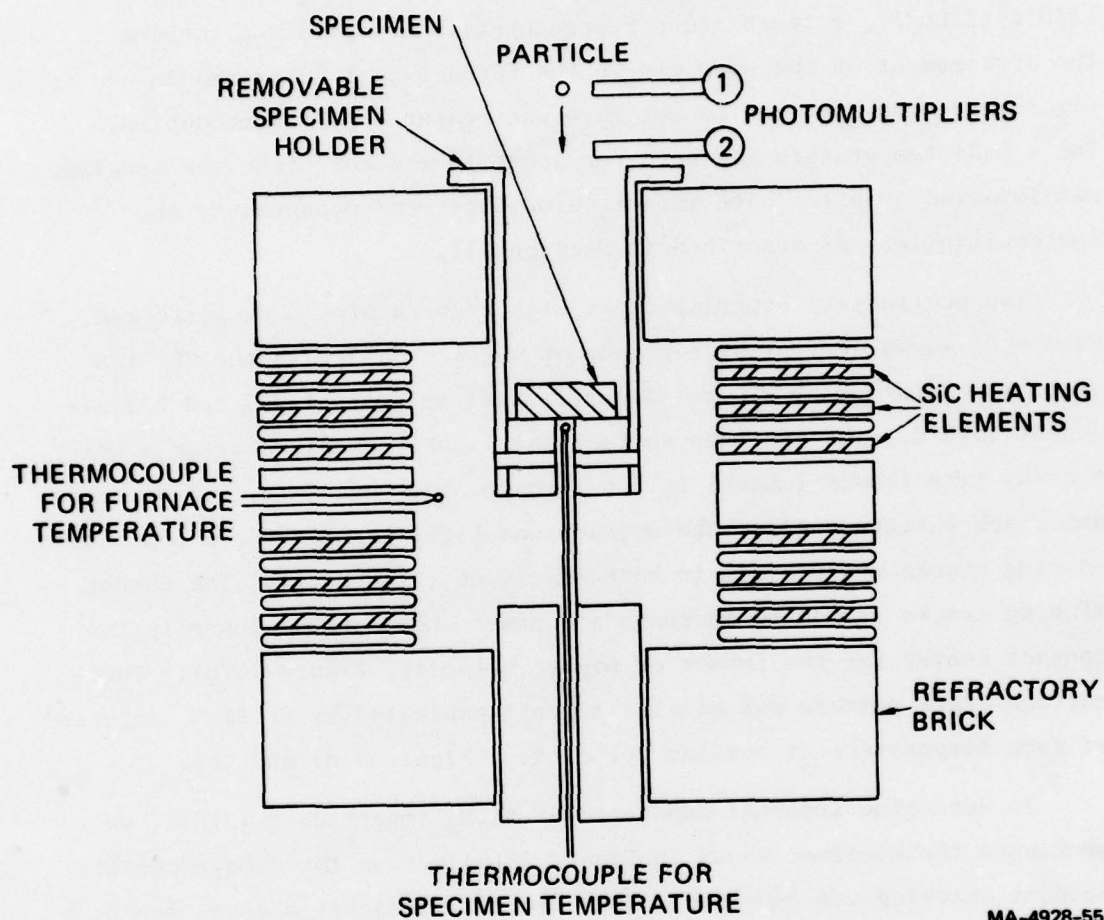
FIGURE 21 TYPICAL QUASI-STATIC INDENTATION DAMAGE IN OXIDIZED HP Si_3N_4 BY 2.4-mm-DIAMETER WC SPHERES

V PARTICLE IMPACT EXPERIMENTS AT HIGH TEMPERATURES

An electric furnace was used to heat the HP Si_3N_4 specimens to 1370°C (2500°F), a temperature representative of operating turbines. The arrangement of the specimen in the furnace is illustrated in Figure 22. The specimen temperature was measured by thermocouples. The steady temperature was held for about 10 minutes, then the specimen was impacted in situ. The impact velocities were measured by the photomultipliers as described in Section III.

Two preliminary experiments at high temperatures were performed. Figure 23 shows optical micrographs of surface damage in the HP Si_3N_4 specimens impacted by 2.4-mm-diameter steel spheres at 127 and 222 m/s and at 1370°C . The specimen surface was lightly oxidized after a brief heat-up time (about 1 hour) in the furnace and, therefore, did not show any crack pattern. After the surface was lightly polished, a population of ring cracks was visible in both specimens (Figure 24). The number of ring cracks seemed to increase and newer ones formed closer to the contact center for the impact at higher velocity, Figure 24(b). The surface crack pattern was similar to that exhibited by HP Si_3N_4 impacted at room temperature at similar velocities, Figure 6(d) and (e).

To determine internal damage to HP Si_3N_4 impacted at 1370°C , we sectioned the specimen shown in Figure 24(b) across the damage center. No cone cracking was observed. This seems to indicate a deviation from the room-temperature fracture process in which subsurface growth of cone cracks with velocity was observed (Figure 10). More high-temperature impacts are necessary to confirm these observations for any reliable evaluation.

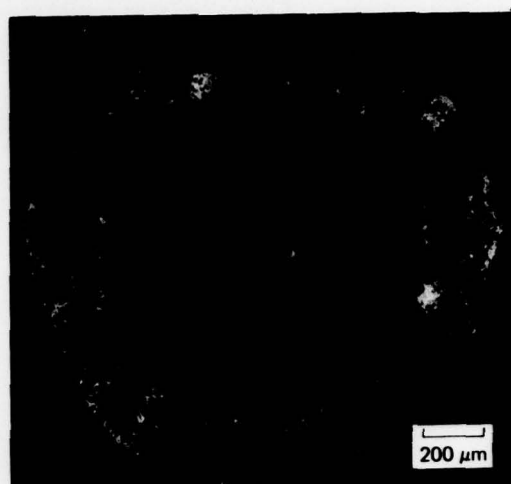


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FIGURE 22 EXPERIMENTAL ARRANGEMENT FOR HIGH-TEMPERATURE PARTICLE-IMPACT TESTS



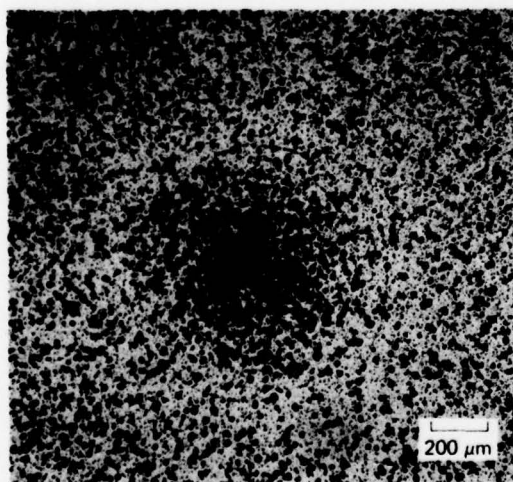
(a) 192 m/s



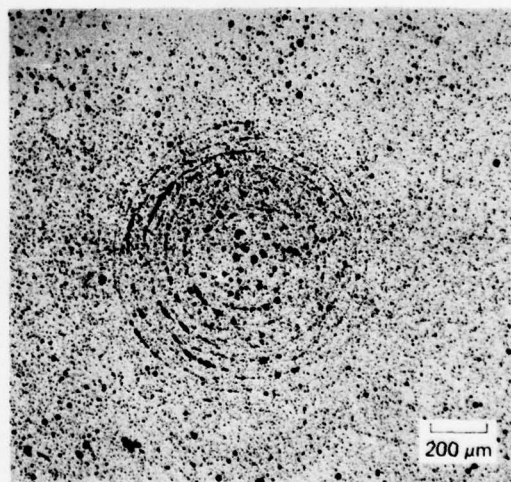
(b) 222 m/s

MP-4928-56

FIGURE 23 SURFACE DAMAGE IN HP Si_3N_4 HEATED TO 1370°C AND IMPACTED WITH 2.4-mm-DIAMETER STEEL SPHERES



(a) 192 m/s



(b) 222 m/s

MP-4928-57

FIGURE 24 RING CRACKS THAT APPEARED AFTER THE SURFACES OF SPECIMENS IN FIGURE 23 WERE LIGHTLY POLISHED, SHOWING FRACTURE DAMAGE IN THE SUBSTRATE BENEATH THE OXIDE

VI EFFECT OF MICROSTRUCTURE ON CRACK PROPAGATION

Ring cracks were produced in HP Si_3N_4 and in RB Si_3N_4 by quasi-static indentation and were examined by replica electron microscopy to determine whether the microstructure influenced crack propagation. Impressions were made with a 2.4-mm-diameter WC sphere using low loads to generate only a relatively small amount of damage in the form of partial ring cracks. For HP Si_3N_4 , it was found that the type of damage produced depended on surface preparation. Ring cracks were seen only on unetched surfaces; plastic impressions with no ring cracks were formed on etched surfaces. Such an effect could arise if thin oxide layers were produced during etching. For the replica studies described here, impressions were made on polished surfaces, with very light etching following indentation. Ring cracks produced dynamically were also examined to determine the effect of microstructure on crack propagation in particle impacts.

Hot Pressed Silicon Nitride

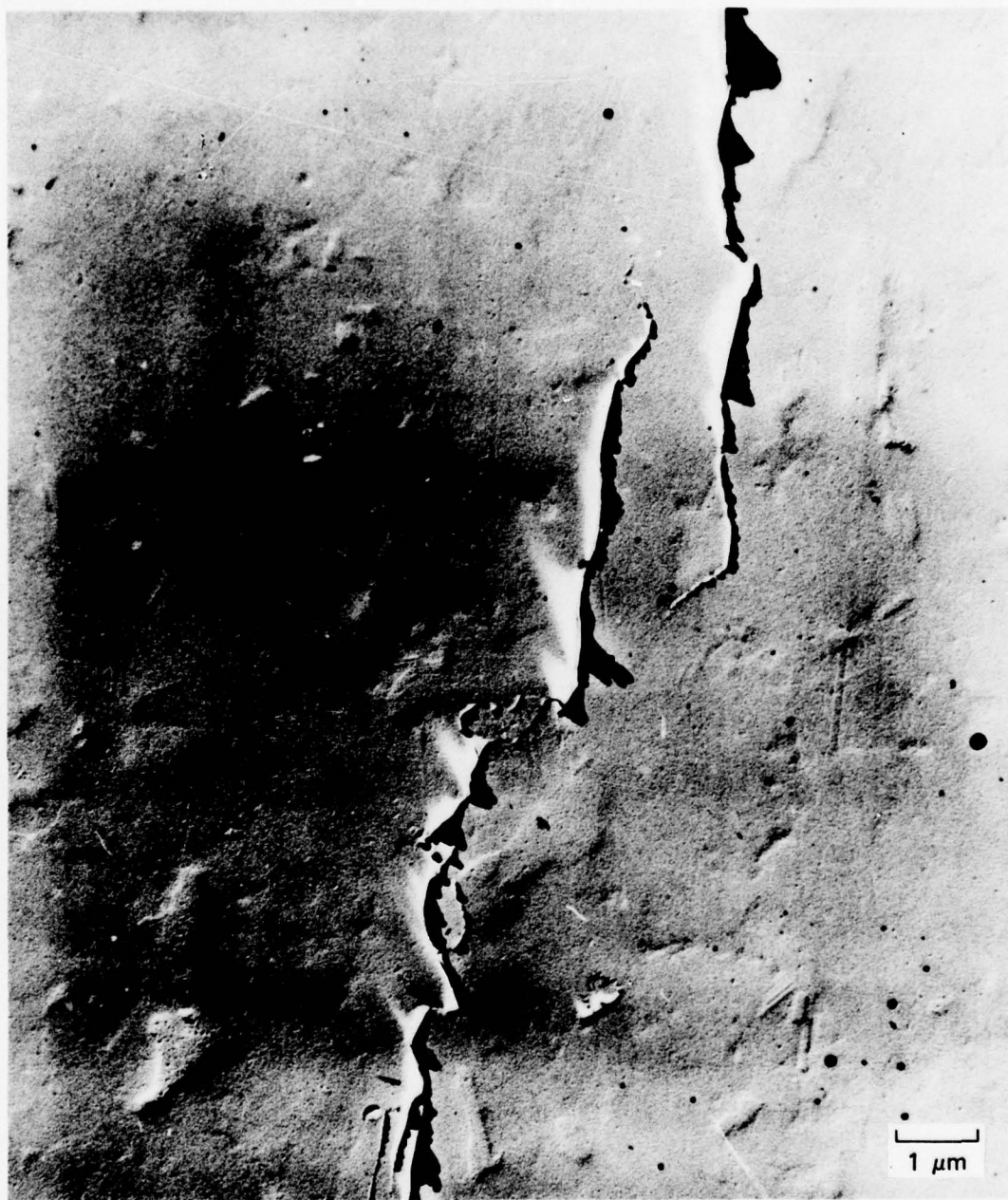
In hot pressed Si_3N_4 the direction of the ring cracks is locally perturbed by the microstructure as illustrated in Figure 25(a). The effect of the microstructure is significant since in some instances the cracks appear to be forced into a radial orientation that may cause them to stop or to form concentric segments of ring cracks as shown in Figure 25(b). The general appearance of cracks indicates intergranular failure. This can be seen more clearly in Figure 26, which also shows that intergranular microfracture occurs in the immediate vicinity of the main propagating crack. These observations are important because they suggest that resistance to impact cracking might be improved by modifying the size and shape of the grains in hot pressed Si_3N_4 .



(a)

MP-4928-75

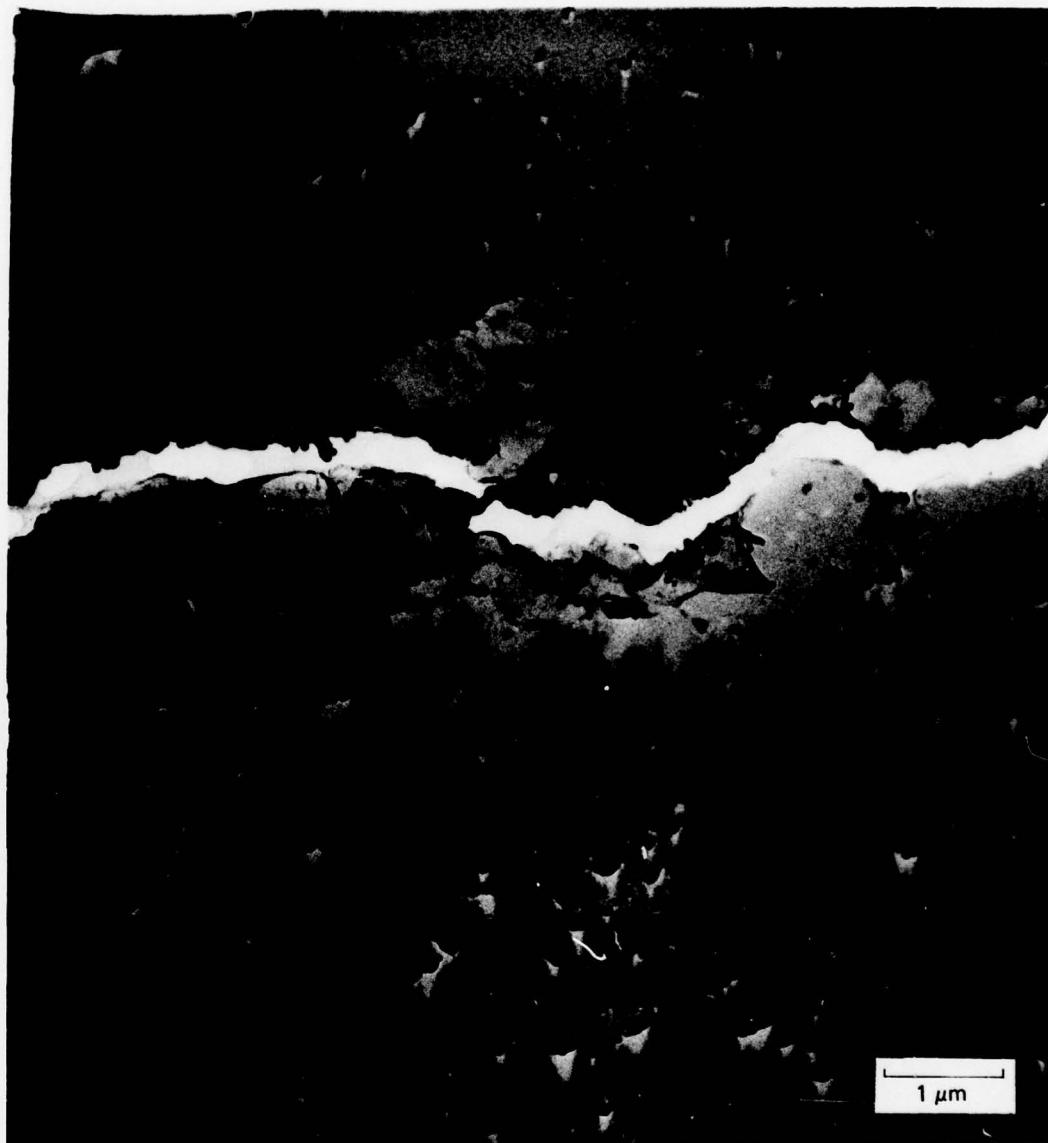
FIGURE 25 TYPICAL RING CRACKS IN HP Si_3N_4 PRODUCED BY QUASI-STATIC INDENTATION



(D)

MP-4928-76

FIGURE 25 TYPICAL RING CRACKS IN HP Si_3N_4 PRODUCED BY QUASI-STATIC INDENTATION (Concluded)



MP-4928-77

FIGURE 26 INTERGRANULAR PROPAGATION OF A RING CRACK IN HP Si₃N₄

Reaction Bonded Silicon Nitride

Examples of ring cracks in RB Si_3N_4 are shown in Figures 27 through 29. The ring cracks follow the imposed Hertzian stress field much more closely than those in HP Si_3N_4 and appear to be diverted only by large pores. Figure 27 shows parts of several concentric ring cracks and an interconnecting crack in radial orientation, which are typical features of damage in RB Si_3N_4 . The interconnecting cracks form when a propagating ring crack is diverted, presumably by a microstructural feature. The propagation direction of these radial segments, relative to the indent center, is not known, but in many cases where the radial segments end in uncracked material, they have propagated toward the center of indentation, as can be seen in Figure 28. Ring cracks also interact with, and occasionally terminate at, large pores as shown in Figure 29.

Samples of ring cracks produced in particle impacts on RB Si_3N_4 are shown in Figures 30 and 31. The general appearance of the ring cracks produced quasi-statically and dynamically is very similar (compare Figures 27 and 30). The effect of the microstructure also appears to be similar. Ring cracks produced in particle impacts are seen to be similarly diverted by large pores and often terminated in them (Figure 31). The grains and the majority of pores in RB Si_3N_4 are smaller than $1\text{ }\mu\text{m}$, however, and do not appear to exert a marked influence on crack propagation and macrofracture.



MP-4928-78

FIGURE 27 TYPICAL RING CRACKING BY QUASI-STATIC INDENTATION IN RB Si_3N_4 : A
Population of Concentric Ring Cracks and Interconnecting Radial Segments



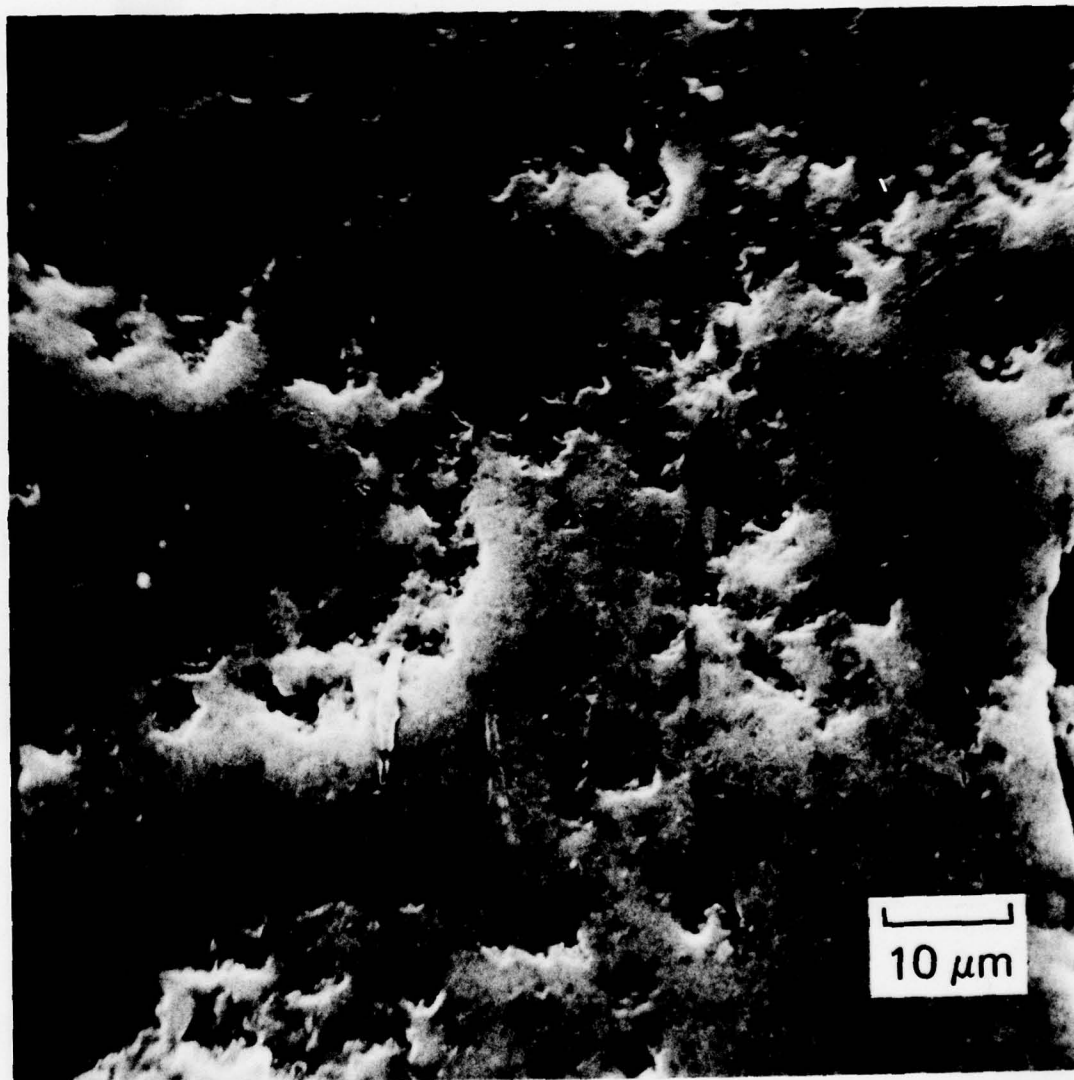
MP-4928-79

FIGURE 28 CRACK TIP OF A RADIAL SEGMENT (Shown in Figure 27) THAT VEERS TOWARD CONTACT CENTER



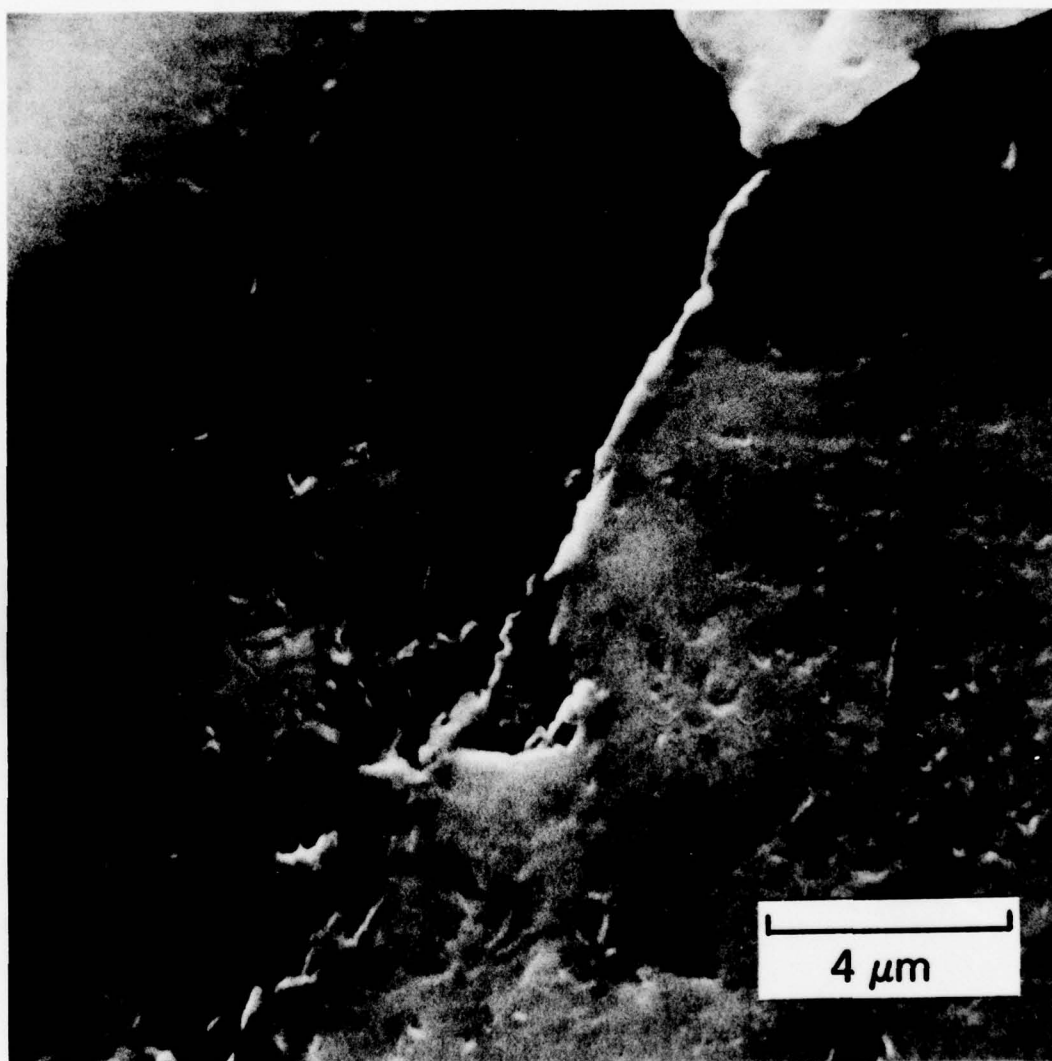
MP-4928-80

FIGURE 29 A RING CRACK IN RB Si_3N_4 PRODUCED BY QUASI-STATIC INDENTATION THAT TERMINATES AT A LARGE PORE



MP-4928-81

FIGURE 30 TYPICAL RING CRACKING BY PARTICLE IMPACT IN RB Si_3N_4



MP-4928-82

FIGURE 31 A RING CRACK IN RB Si₃N₄ PRODUCED BY PARTICLE IMPACT THAT TERMINATES AT A LARGE PORE

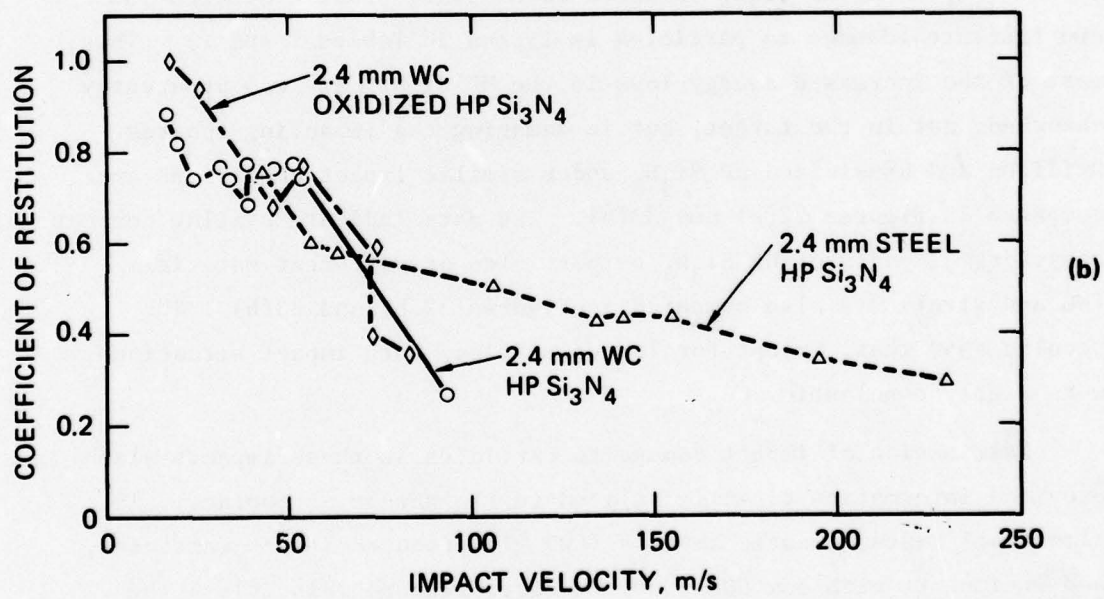
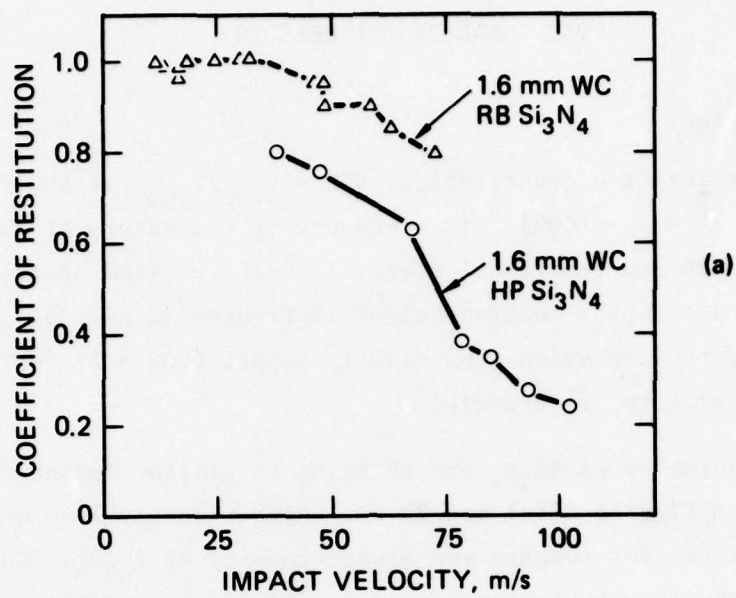
VII ANALYSIS OF RESULTS

Nature of Contact

The coefficient of restitution, $COR = V_{reb}/V_{imp}$, or the fractional energy loss, $\frac{\Delta E}{E} = 1 - (COR)^2$, is a measure of the nature of contact. Experimental COR and fractional energy loss are plotted against impact velocity for all impact configurations in Figures 32 and 33. For all impact cases, the deviation from elastic impact ($COR = 1$) increased with impact velocity, as expected.

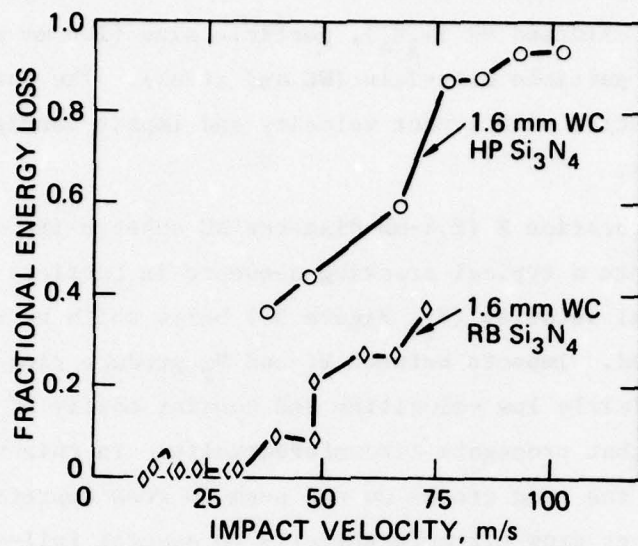
The behavior of RB Si_3N_4 and HP Si_3N_4 in similar impact conditions is compared in Figures 32(a) and 33(a). Higher energy loss and lower COR are indicated for tougher and higher-density HP Si_3N_4 . Surface damage is heavy in both target materials (Figures 8 and 13), but WC spheres impacting HP Si_3N_4 suffered more severe plastic deformation and fracture (damage to particles is listed in Tables 1 and 2). Thus, most of the increased energy loss in the HP Si_3N_4 case was apparently absorbed, not in the target, but in damaging the impacting spheres. Oxidized and unoxidized HP Si_3N_4 under similar impact conditions are compared in Figures 32(b) and 33(b). The data indicate similar contact behavior. Impacts of HP Si_3N_4 by particles of different materials (WC and steel) are also compared in Figures 32(b) and 33(b). The results show that, except for low velocities, both impact situations were highly nonelastic.

Examination of impact damage to particles in these impacts also provided information directly related to the nature of contact. In almost all cases, elastic impacts ($COR \approx 1$) produced intact particles, and in impacts with low COR (i.e., reduced rebound velocities) the particles were heavily deformed or fragmented. Observed particle damage for different tests is listed with other impact parameters in Tables 1 through 3.

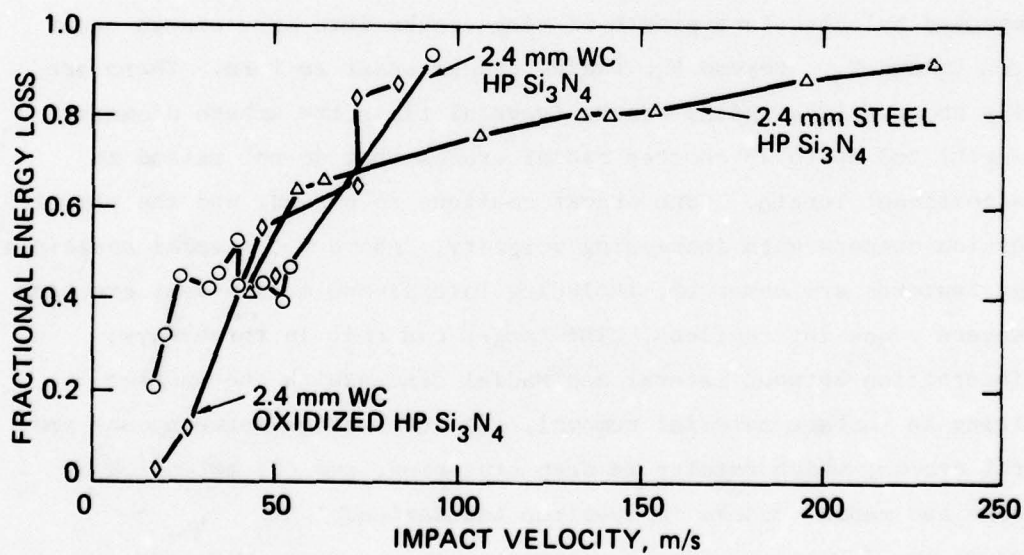


MA-4928-58

FIGURE 32 COEFFICIENT OF RESTITUTION



(a)



(b)

MA-4928-59

FIGURE 33 FRACTIONAL ENERGY LOSS

Failure Modes

The fractographic examinations of individual impact configurations described in Sections III and V are now combined for an overall study of the specimen failure modes as influenced by various target materials (HP and RB Si_3N_4 , oxidized HP Si_3N_4), particle size (1.6 mm and 2.4 mm in diameter), and particle materials (WC and steel). The variation of damage characteristics with impact velocity and impact configuration is given in Figure 34.

Impact configuration B (2.4-mm-diameter WC spheres impacting HP Si_3N_4) represents a typical cracking sequence in particle impacts. There is a critical velocity (V_1 , Figure 34) below which no target damage was observed. Impacts between V_1 and V_2 produce ring cracks that nucleate at fairly low velocities and consist mostly of short segmental cracks that propagate circumferentially. In this velocity range (V_1 to V_2), the ring cracks do not seem to grow appreciably in depth; instead, they grow circumferentially to several full-circle cracks as the velocity increases. Surface plastic flow (plastic impressions) accompanied by subsurface growth of ring cracks into cone cracks occur between V_2 and V_3 . Beyond V_3 , radial cracks start to form. There are usually about 9 long radial cracks (several times the sphere diameter in length) and up to 25 shorter radial cracks that do not extend to any significant length. Cone cracks continue to extend, and the plastic impression deepens with increasing velocity. Above V_4 , several additional damage features are observed, including lateral and median-vent cracking and severe crack interactions. The target can fail in three ways: (1) interaction between lateral and radial cracks with the surface resulting in surface material removal, (2) interaction between cone and lateral cracks, which results in deep cratering, and (3) extension of the cone and radial cracks to specimen boundaries.

Response of RB and HP Si_3N_4 to similar impacts can be compared in Figure 34 (configurations D and C). The cracking sequence is similar for both materials, but all stages of fracture development in RB Si_3N_4 occur in a much narrower range and at substantially lower impact velocity.

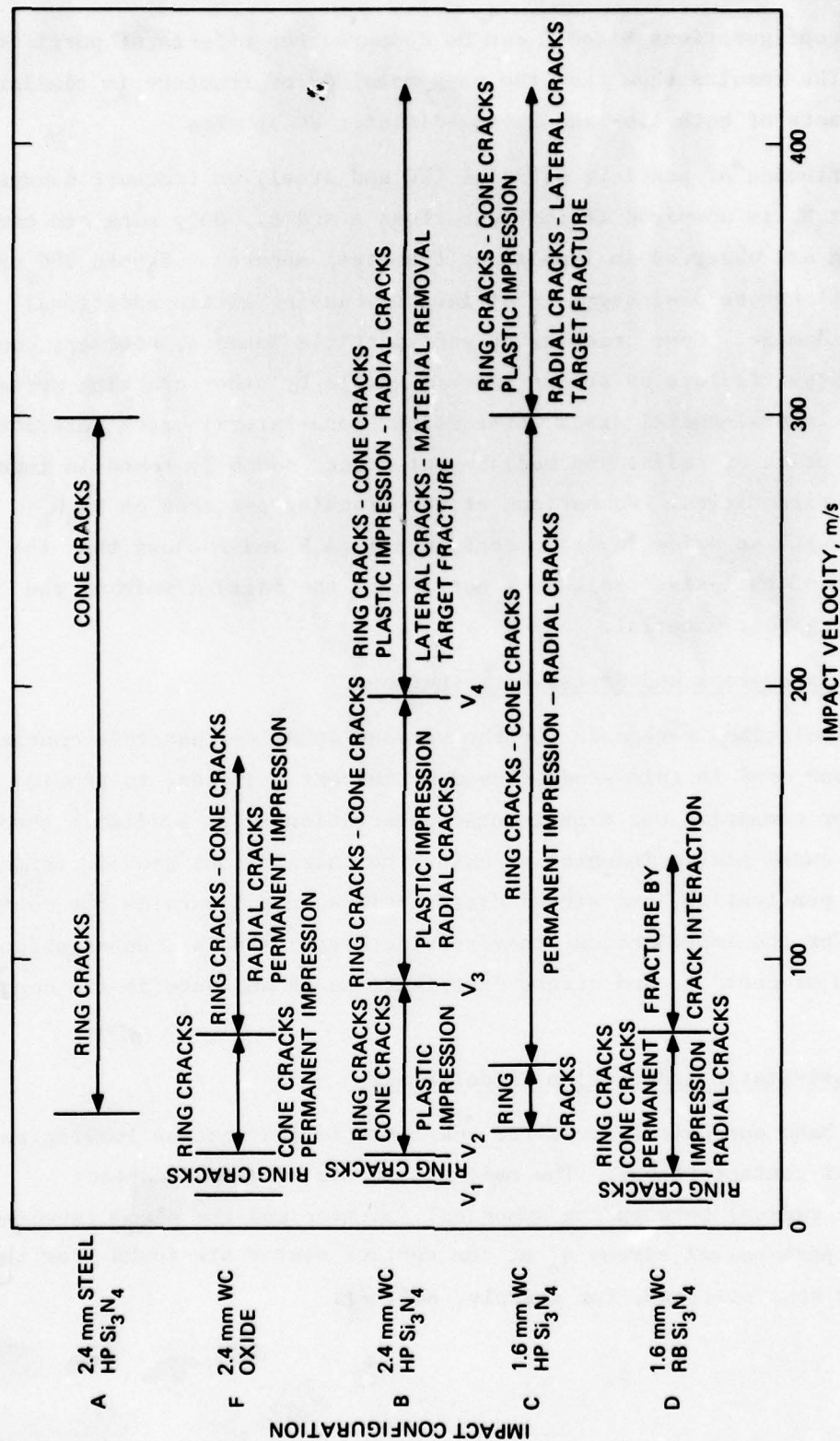


FIGURE 34 SPECIMEN FAILURE MODES

Impact configurations B and C can be compared for effects of particle size. The results show that the phenomenology of fracture is similar for impacts of both 1.6- and 2.4-mm-diameter WC spheres.

Influence of particle material (WC and steel) on fracture damage in HP Si_3N_4 is compared in configurations A and B. Only ring and cone cracking are observed in impacts by the steel spheres. Beyond 300 m/s, the steel sphere disintegrates at impact, causing little additional surface damage. Cone cracking by soft-particle impacts, however, could cause target failure as severe as that caused by other cracking mechanisms such as lateral-radial crack interaction, cone-lateral crack interaction, and extension of radial and median-vent cracks commonly found in impacts by harder particles. Comparison of the cracking patterns on Si_3N_4 covered with an oxide layer in configurations B and F shows that the presence of the oxide scale does not change the failure mode of the underlying host material.

Contact Parameters and Stress Distributions

Calculations were made for the various spherical particle contact situations used in this study, based on current analyses, to provide a means for comparing our experimental observations with available theory. For the quasi-static indentation case, the calculations provide contact radius, penetration, and stress distributions in and outside the contact zone. For the impact cases, they provide contact radius, penetration, duration of contact, and stress distributions in and outside the contact zone.

Quasi-Static Indentation Calculations

We base our calculations for quasi-static indentation loading on the Hertz contact theory. The radius a of the circle of contact (contact radius) between the spherical indenter and the plane (specimen) and the peak normal stress q_0 at the contact center are found from the Hertzian analysis (see, for example, Ref. 7):

$$a = \left[\frac{3FR}{4} \left(\frac{1 - \nu_1^2}{E_1} + \frac{1 - \nu_2^2}{E_2} \right) \right]^{1/3} \quad (1)$$

$$q_o = \frac{3F}{2\pi a^2} \frac{3F^{1/3}}{2\pi \left[\frac{3R}{4} \left(\frac{1 - \nu_1^2}{E_1} + \frac{1 - \nu_2^2}{E_2} \right) \right]^{2/3}} \quad (2)$$

where F is the normal load on the indenter, R is the indenter radius, E_1 and E_2 are the Young's moduli of the plane and indenter, respectively, and ν_1 and ν_2 are the corresponding values of the Poisson's ratio.

A force-penetration relation can also be obtained from the Hertz contact theory:

$$F = \frac{4X^{3/2} R^{1/2}}{3 \left(\frac{1 - \nu_1^2}{E_1} + \frac{1 - \nu_2^2}{E_2} \right)} \quad (3)$$

where X is the penetration or distance of mutual approach.

The normal stress between a spherical indenter and a plane is shown by the Hertz contact theory to vary from q_o at the center to zero at the outer edge of the contact area.

$$q = \frac{q_o}{a} \sqrt{a^2 - r^2} \quad (4)$$

where r is the radial distance from the center of the contact area. Because of this normal stress, large radial and circumferential (hoop) stresses occur at the surface of the plate. The amplitude of the radial and circumferential stresses at the surface of an elastic plate will now be determined for statically applied spherical indenter.

The solution procedure follows the analysis of Bycroft⁸ for loadings on an elastic half-space. First the radial and vertical displacements are written as integrals of Bessel functions.

$$u = \int_0^{\infty} (A_1 + B_1 z) e^{-xz} J_1(xr) dx \quad (5)$$

$$w = \int_0^{\infty} \left[A_1 - \frac{B_1(\tau^2 + 1)}{x(\tau^2 - 1)} + B_1 z \right] e^{-xz} J_0(xr) dx \quad (6)$$

where A_1 and B_1 = functions of x , the variable of integration

r = radial coordinate

z = depth coordinate

$\tau^2 = \mu/(\lambda + 2\mu)$

λ, μ = Lamé's constants.

Next we use the elastic stress-strain relations (specialized for the axisymmetric case) to determine the radial, depth, hoop, and shear stresses

$$\sigma_{rr} = \lambda \left(\frac{\partial u}{\partial r} + \frac{u}{r} + \frac{\partial w}{\partial z} \right) + 2\mu \frac{\partial u}{\partial r} \quad (7)$$

$$\sigma_{zz} = \lambda \left(\frac{\partial u}{\partial r} + \frac{u}{r} + \frac{\partial w}{\partial z} \right) + 2\mu \frac{\partial w}{\partial z} \quad (8)$$

$$\sigma_{\theta\theta} = \lambda \left(\frac{\partial u}{\partial r} + \frac{u}{r} + \frac{\partial w}{\partial z} \right) + 2\mu \frac{u}{r} \quad (9)$$

$$\sigma_{rz} = \mu \left(\frac{\partial u}{\partial z} + \frac{\partial w}{\partial r} \right) \quad (10)$$

The shear stress expression at the surface ($z = 0$) is obtained by inserting the integrals from Eqs. (5) and (6) into Eq. (10):

$$\sigma_{rz} = \int_0^{\infty} 2\mu \left[\frac{B_1 \tau^2}{\tau^2 - 1} - A_1 x \right] J_1(xr) dx \quad (11)$$

A zero shear stress condition on the surface is obtained by setting

$$\frac{B_1 \tau^2}{\tau^2 - 1} = A_1 x = A_2 \tau^2 x \quad (12)$$

where A_2 is a new function of x . Next we obtain the expression for stress in the depth direction at the surface.

$$\begin{aligned} \sigma_{zz} &= \int_0^\infty 2\mu \left[\frac{B_1}{\tau^2 - 1} - A_1 x \right] J_0(xr) dx \\ &= 2\mu(\tau^2 - 1) \int_0^\infty A_2(x) x J_0(xr) dx \end{aligned} \quad (13)$$

In this equation we have introduced the new variable A_2 from Eq. (12). Now we wish to evaluate A_2 such that σ_{zz} will equal the applied stress in Eq. (4) in the region of contact and be zero elsewhere on the surface. A_2 is obtained by equating the loading to a Fourier-Bessel expansion as follows:

$$\begin{aligned} q &= \frac{q_0}{a} \sqrt{a^2 - r^2} = \frac{q_0}{a} \int_0^\infty J_0(xr) x \left[\int_0^\infty \sqrt{a^2 - t^2} J_0(xt) t dt \right] dx \\ &= \frac{q_0}{2} \frac{2\pi a}{2} \int_0^\infty J_{3/2}(xa) J_0(xr) x^{-3/2} dx \end{aligned} \quad (14)$$

where the integration has been performed with Sonine's first finite integral (p. 740 of Ref. 9 and p. 406 of Ref. 10). By equating Eqs. (13) and (14), we now evaluate A_2

$$A_x(x) = \frac{q_0}{4\mu(\tau^2 - 1)} \frac{2\pi a J_{3/2}(xa)}{x^{3/2}} \quad (15)$$

Next the expression for σ_{rr} , Eq. (7), is derived from the equations for u and w , Eqs. (5) and (6) and then evaluated at the surface where $z = 0$. The result is

$$\begin{aligned}\sigma_{rr} &= 2(\lambda + \mu) \int_0^\infty A_1(x) x J_0(xr) dx - \frac{2\mu}{r} \int_0^\infty A_1(x) J_1(xr) dx \\ &= \frac{q_0^2 \sqrt{2\pi a}}{2\mu(\tau^2 - 1)} \left[(\lambda + \mu) \int_0^\infty J_{3/2}(xa) J_0(xr) x^{-1/2} dx \right. \\ &\quad \left. - \frac{\mu}{r} \int_0^\infty J_{3/2}(xa) J_1(xr) x^{-3/2} dx \right]\end{aligned}\quad (16)$$

These integrals are found in standard tables such as Gradshteyn and Ryzhik⁹. For $r < a$, that is, within the contact region, the radial stress is

$$\sigma_{rr} = \frac{q_0 \sqrt{\pi}}{2\Gamma(3/2)} \left[F\left(1, -\frac{1}{2}, 1, \frac{r^2}{a^2}\right) - \frac{\mu}{\lambda + \mu} \frac{\Gamma(1)}{2\Gamma(2)} F\left(1, -\frac{1}{2}, 2, \frac{r^2}{a^2}\right) \right] \quad (17)$$

where F is the hypergeometric function and Γ is the gamma function. At the boundary of the contact area, $r = a$, and the radial stress is

$$\sigma_{rr} = \frac{q_0 \sqrt{\pi}}{2} \left[\frac{\Gamma(\frac{1}{2}) \Gamma(1)}{\Gamma(0) \Gamma(\frac{3}{2}) \Gamma(\frac{3}{2})} - \frac{\mu}{\lambda + \mu} \cdot \frac{1}{2\Gamma(\frac{5}{2})} \right] \quad (18)$$

Outside the contact area, where $r > a$, the radial stress is

$$\begin{aligned}\sigma_{rr} &= \frac{q_0 \sqrt{\pi a^2}}{2r^2} \left[\frac{\Gamma(1) F(1, 1, \frac{5}{2}, \frac{a^2}{r^2})}{\Gamma(0) \Gamma(\frac{5}{2})} \right. \\ &\quad \left. - \frac{\mu}{(\lambda + \mu) 2 \Gamma(\frac{5}{2})} F(1, 0, \frac{5}{2}, \frac{a^2}{r^2}) \right]\end{aligned}\quad (19)$$

The three expressions for radial stress can be reduced somewhat by evaluating the Gamma functions (Ref. 10, p. 3) and writing the hypergeometric functions as integrals (Ref. 11, p. 59). The following intermediate results are obtained.

$$F(1, -\frac{1}{2}, 1, \frac{r^2}{a^2}) = \sqrt{1 - r^2/a^2}$$

$$F(1, -\frac{1}{2}, 2, \frac{r^2}{a^2}) = -\frac{2a^2}{3r^2} \left[\left(1 - \frac{r^2}{a^2}\right)^{3/2} - 1 \right]$$

$$F(1, 1, \frac{5}{2}, \frac{a^2}{r^2}) = \frac{3r^2}{a^2} \left[1 - \sqrt{\frac{r^2 - a^2}{a^2}} \operatorname{arctan} \sqrt{\frac{a^2}{r^2 - a^2}} \right]$$

$$F(1, 0, \frac{5}{2}, \frac{a^2}{r^2}) = 1$$

$$\Gamma(0) = \infty$$

$$\Gamma(\frac{1}{2}) = \sqrt{\pi}$$

$$\Gamma(1) = 1$$

$$\Gamma(\frac{3}{2}) = \sqrt{\pi}/2$$

$$\Gamma(2) = 1$$

$$\Delta(\frac{5}{2}) = 3\sqrt{\pi/4}$$

In Eqs. (17) through (19), the ratio $\mu/(\lambda + \mu)$ is replaced by $1 - 2\nu$, where ν is Poisson's ratio. Then these equations become

For $r < a$:

$$\sigma_{rr} = q_0 \left\{ \sqrt{1 - r^2/a^2} + (1 - 2\nu) \frac{a^2}{3r^2} [(1 - r^2/a^2)^{3/2} - 1] \right\} \quad (20)$$

For $r = a$:

$$\sigma_{rr} = - \frac{(1 - 2\nu)}{3} q_0 \quad (21)$$

For $r > a$:

$$\sigma_{rr} = - \frac{(1 - 2\nu)}{3} q_0 \cdot \frac{a^2}{e^2} \quad (22)$$

For $r = 0$, Eq. (20) becomes

$$\sigma_{rr} = \frac{(1 + 2\nu)}{2} q_0 \quad (23)$$

With the radial stress known, the circumferential stress $\sigma_{\theta\theta}$ can be easily found. Starting with Eq. (9), the expressions for u and w , and the value of A_2 from Eq. (15), we obtain an expression for $\sigma_{\theta\theta}$ comparable to Eq. (16)

$$\sigma_{\theta\theta} = 2\lambda \int_0^\infty A_1 \times J_0(xr) dx + \frac{2\mu}{r} \int_0^\infty A_1 J_1(xr) dx \quad (24)$$

These two terms are proportional to the corresponding terms for σ_{rr} . We need only multiply the first term in σ_{rr} by $\lambda/(\lambda + \mu) = 2\nu$ and change the sign of the second term in σ_{rr} . Then the final results are obtained from Eqs. (20) through (22).

For $r < a$:

$$\sigma_{\theta\theta} = q_0 \left\{ 2\nu \sqrt{1 - r^2/a^2} - (1 - 2\nu) \frac{a^2}{3r^2} [(1 - r^2/a^2)^{3/2} - 1] \right\} \quad (25)$$

For $r = a$:

$$\sigma_{\theta\theta} = \frac{1 - 2\nu}{3} q_0 \quad (26)$$

For $r > a$:

$$\sigma_{\theta\theta} = \frac{(1 - 2\nu) q_0 a^2}{3r^2} \quad (27)$$

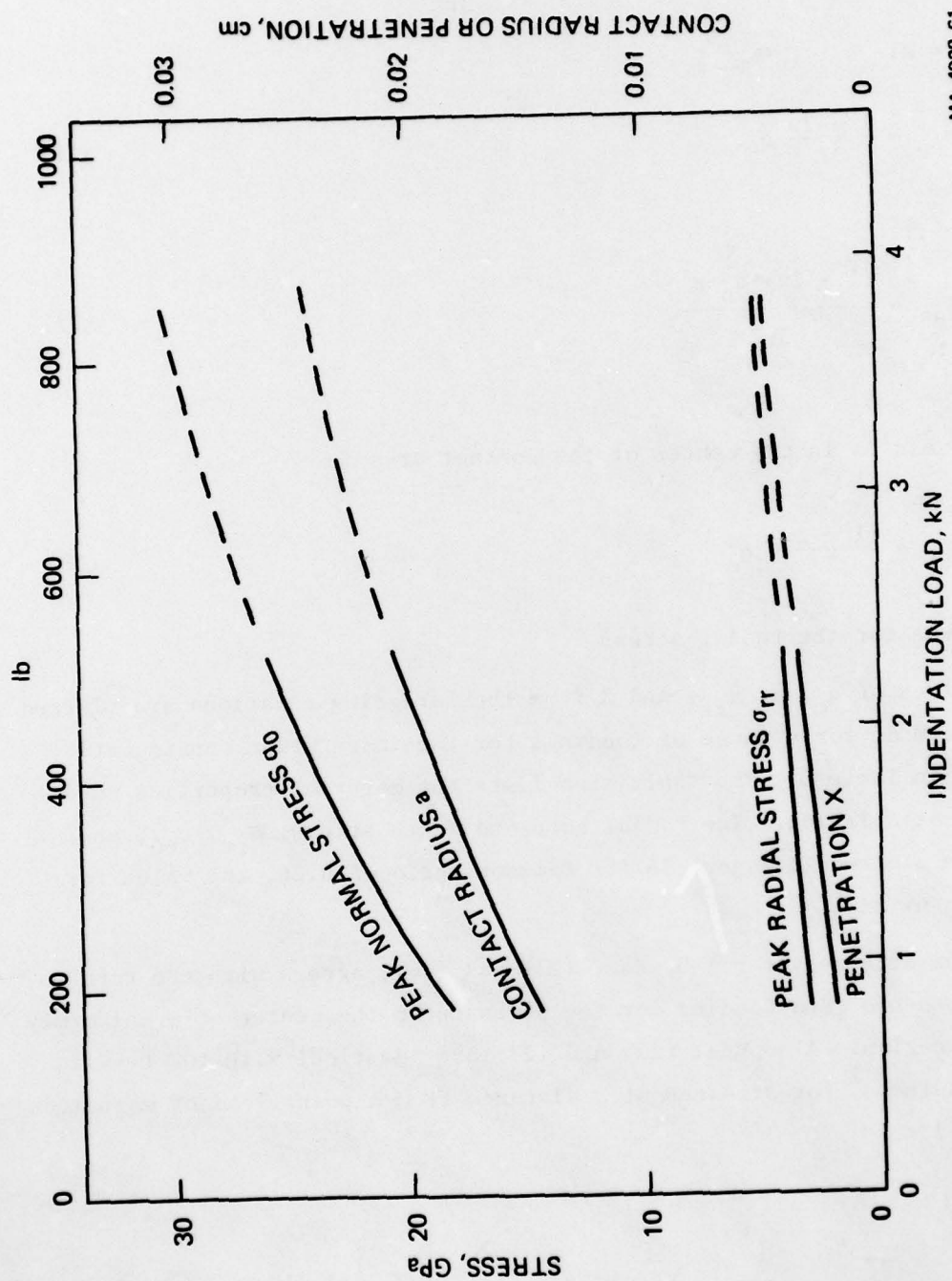
The stress is in the center of the contact area is

$$\sigma_{\theta\theta} = \frac{(1 + 2\nu)}{2} q_0 \quad (28)$$

as it was for the radial stress.

The values of q_0 , a , θ_{rr} , and X from the foregoing equations are plotted in Figure 35 for a range of loadings for the indentation configuration listed in Table 5. The table also lists the material properties used in the calculation. The radial hoop and depth stress, σ_{rr} , $\sigma_{\theta\theta}$, and σ_{zz} are plotted in Figure 36 for Poisson ratio of 0.26, the value for silicon nitride.

The stresses at $r = 0$, Eqs. (23) and (28), agrees with the results of Timoshenko⁷ and Goodier for the stresses in the center of a uniformly loaded region. Also Eqs. (22) and (27) are identical with the results of Boussinesq⁷ for stresses at a distance from a point load of magnitude $2q_0 \pi a^2/3$.



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FIGURE 35 QUASI-STATIC CONTACT PARAMETERS (Indenter: 2.4-mm-Diameter WC Sphere Specimen: HP Si₃N₄)

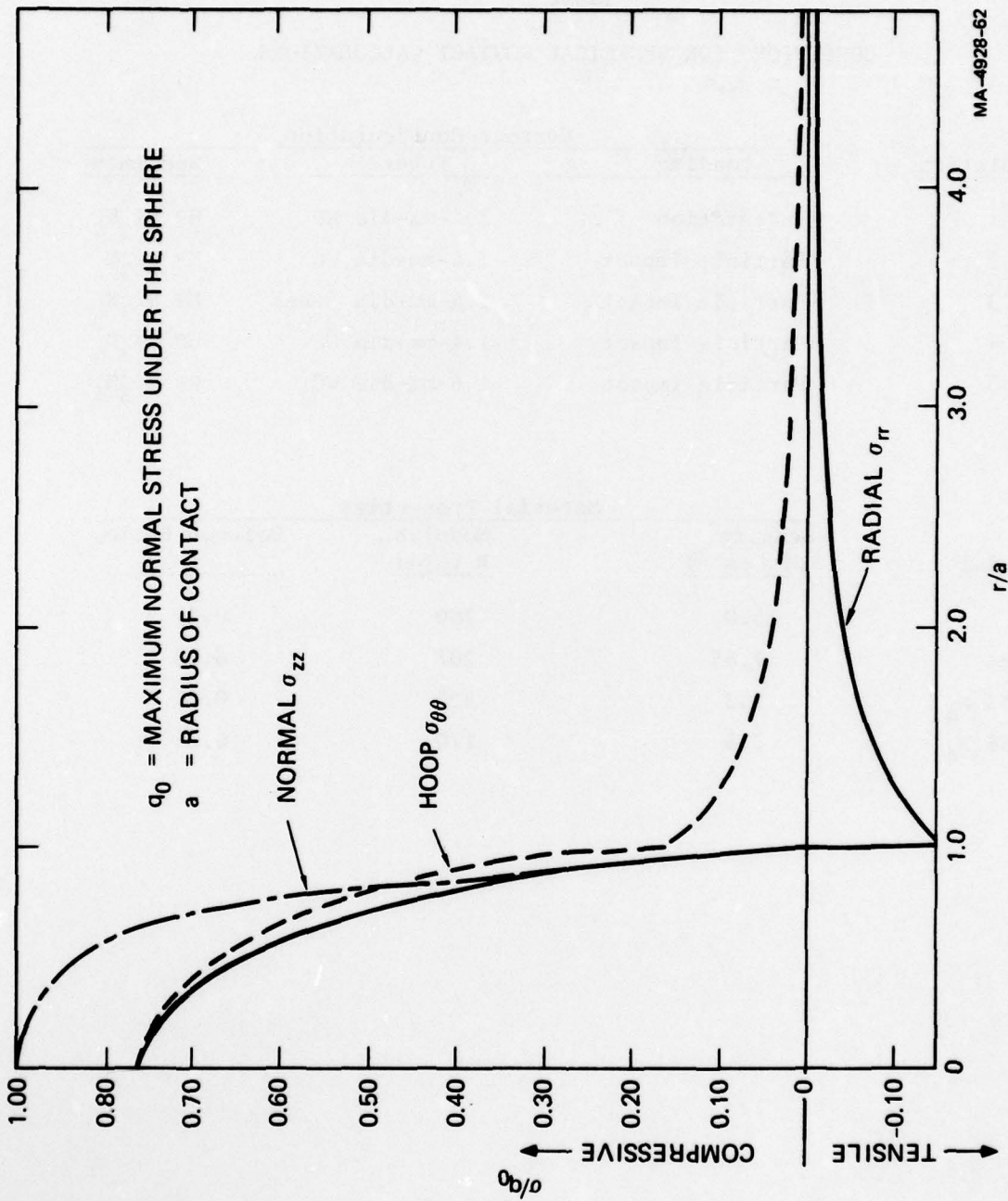
Dotted lines are for range where experiments indicated inelastic behavior of the indenter.

Table 5

CONDITIONS FOR SPHERICAL CONTACT CALCULATIONS

Calculation	Contact Configuration		Specimen
	Loading	Sphere	
1	Indentation	2.4-mm-dia WC	HP Si ₃ N ₄
2	Particle Impact	2.4-mm-dia WC	HP Si ₃ N ₄
3	Particle Impact	2.4-mm-dia steel	HP Si ₃ N ₄
4	Particle Impact	1.6-mm-dia WC	HP Si ₃ N ₄
5	Particle Impact	1.6-mm-dia WC	RB Si ₃ N ₄

Material	Material Properties		
	Density, $\rho(\text{g cm}^{-3})$	Modulus, E (GPa)	Poisson Ratio, ν
WC	15.0	700	0.24
Steel	7.85	207	0.28
HP Si ₃ N ₄	3.3	320	0.26
RB Si ₃ N ₄	2.5	170	0.26



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FIGURE 36 SURFACE STRESS DISTRIBUTIONS FOR QUASI-STATIC AND DYNAMIC ELASTIC CONTACTS

Particle Impact Calculations

An approximate analysis of the spherical particle impact problem was made to provide a means for relating stresses and damage in the impact case to those in the static case. The spherical particle and the impacted plate (half-space) were taken to be elastic. The Hertz contact solution provided the force-displacement relations between the bodies. The present analysis provides the normal stresses in the contact area, extent of the contact area, duration of the impact, and displacement of the sphere after initial contact.

The static elastic stress distribution is used, in the analysis although the problem is dynamic and stresses are above the yield values. Hence the results can be expected to provide only an indication of the stress, duration, and contact area. The effects of the approximations are discussed at the end of this section.

The impact calculation was made by solving the following three equations with a finite difference technique:

$$u = \frac{dX}{dt} \quad (\text{velocity-displacement}) \quad (29)$$

$$F = M \frac{du}{dt} \quad (\text{momentum}) \quad (30)$$

$$F = F(X) \quad (\text{force-displacement}) \quad (31)$$

where F = normal force between the sphere and the plane

M = particle mass

u = particle velocity

X = particle displacement

t = time.

The equations are integrated by writing them in finite-difference form and then integrating them over short time increments to obtain the force, velocity, and displacement history. In finite-difference form, the first two equations are

$$X_i = X_{i-1} + U_{i-1/2} \Delta t_{i-1/2} \quad (32)$$

$$\Delta U_i = \frac{F_i}{M} \Delta t_i \quad (33)$$

where the subscript i refers to the time increment. Hence X_i and F_i pertain to the i^{th} time t_i , and $U_{i-1/2}$ pertains to a time $t_{i-1/2}$. The increments are

$$\Delta U_i = U_{i+1/2} - U_{i-1/2}$$

$$\Delta t_i = t_{i+1/2} - t_{i-1/2}$$

$$\Delta t_{i-1/2} = t_i - t_{i-1}$$

With the noted subscripts in Eq. (32) and (33), the differences are all central differences. Hence all derivatives and other terms pertain to the same time.

The force-displacement relation is obtained from the Hertz contact theory:

$$F = \frac{4X^{3/2} \sqrt{R}}{3 \left(\frac{1 - \nu_1^2}{E_1} + \frac{1 - \nu_2^2}{E_2} \right)} \quad (3)$$

where R is the radius of the sphere

ν_1, ν_2 are Poisson's ratio of plane and sphere

E_1, E_2 are Young's modulus of plane and sphere.

Formulas for radius a of the contact area and the peak stress q_0 are also obtained from the Hertz theory:

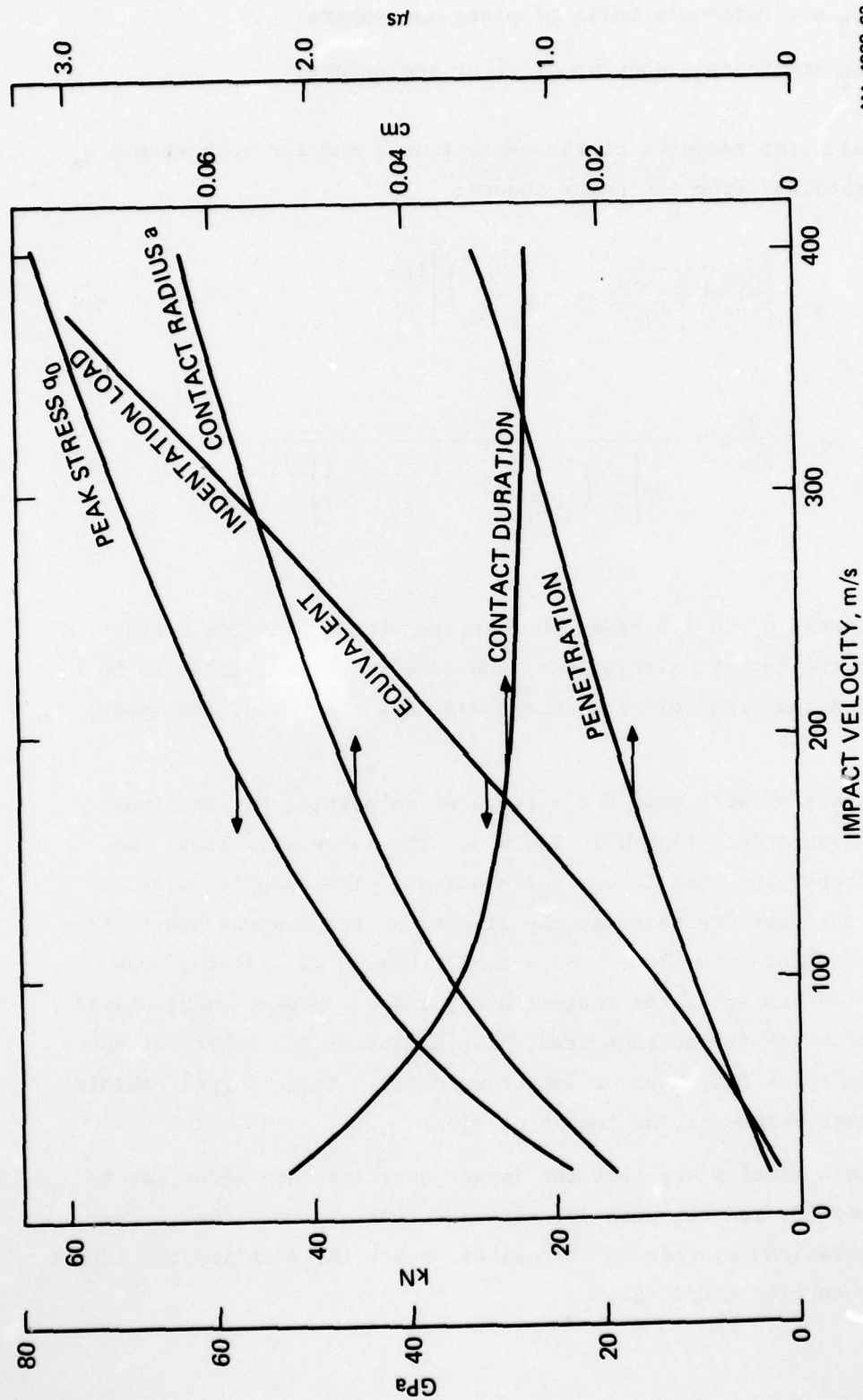
$$a = \left[\frac{3FR}{4} \left(\frac{1 - \nu_1^2}{E_1} + \frac{1 - \nu_2^2}{E_2} \right) \right]^{1/3} \quad (1)$$

$$q_0 = \frac{3F}{2\pi a^2} = \frac{3F^{1/3}}{2\pi \left[\frac{3R}{4} \left(\frac{1 - \nu_1^2}{E_1} + \frac{1 - \nu_2^2}{E_2} \right) \right]^{2/3}} \quad (2)$$

The peak stress q_0 is 1.5 times the average stress over the contact area. For the impact calculations, the time steps were taken to be small enough that the velocity change was less than 1% of the impact velocity.

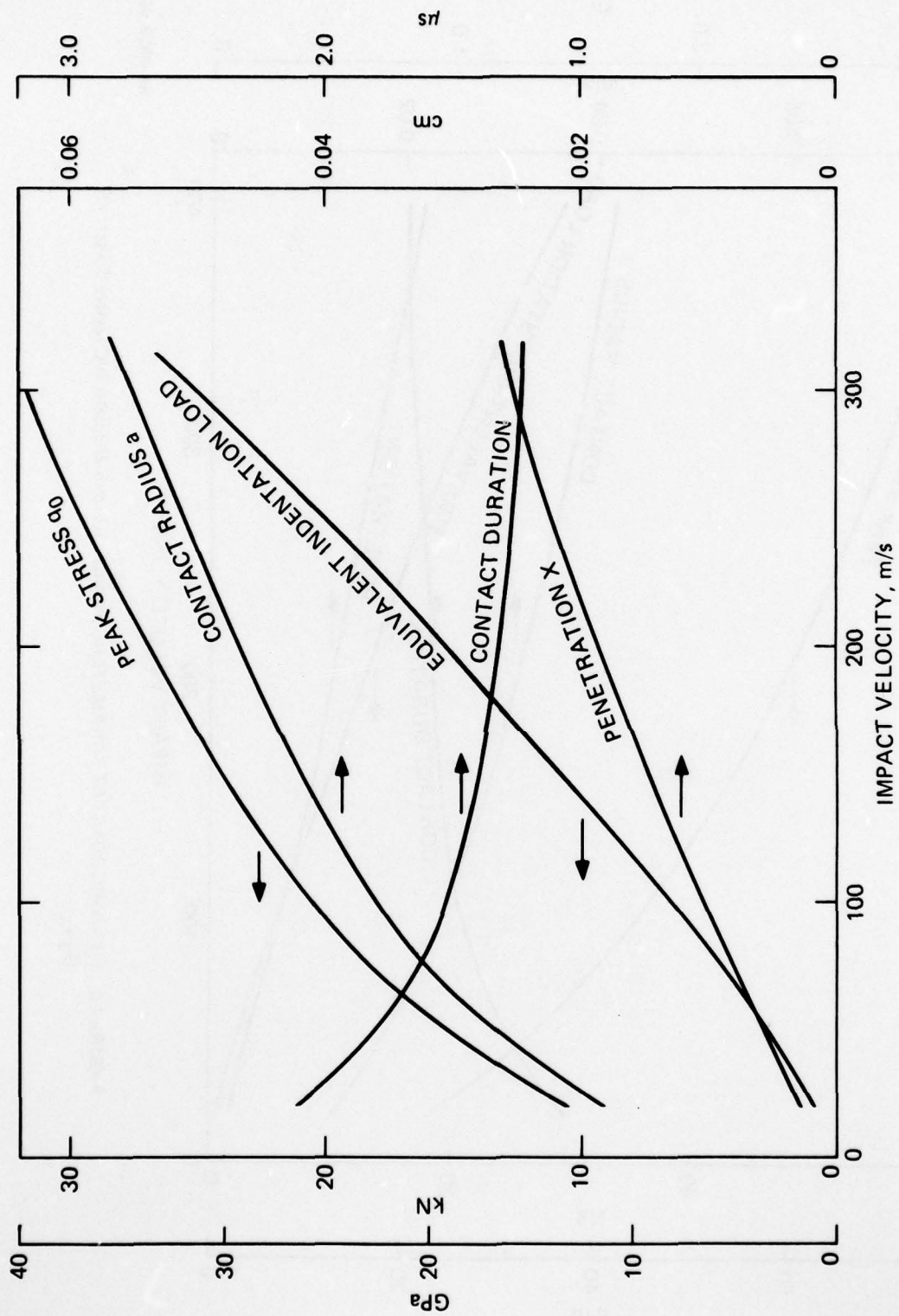
Computations were made for a range of velocities for the four impact configurations listed in Table 5. The table also lists the material properties used in the calculations. The quantities of principal interest are those at the time t_m of the maximum penetration of the sphere into the plane. Figures 37 through 40 plot the peak stress at the center of the contact area, the equivalent quasi-static load F , radius of the contact area, displacement of the sphere at the time t_m and t_m as functions of impact velocity. These curves exhibit the nonlinear nature of the impact problem.

The main results are that the impact durations are about one to two microseconds and the peak stresses are tens of GPa. The force-velocity relations provide an approximate means for relating the impact and static testing conditions.



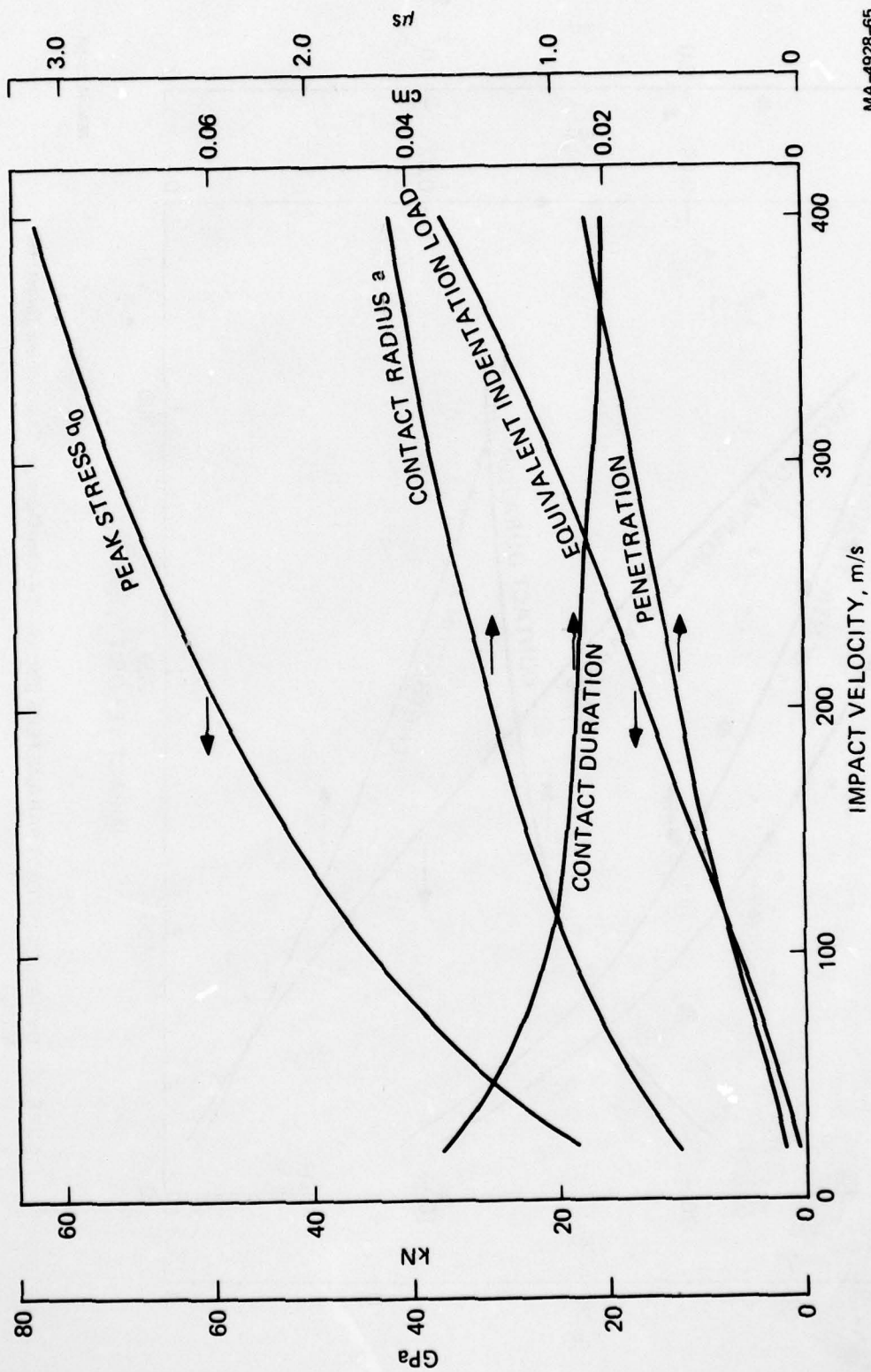
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FIGURE 37 DYNAMIC CONTACT PARAMETERS (Particle: 2.4-mm-Diameter WC Sphere
Target: HP Si₃N₄)



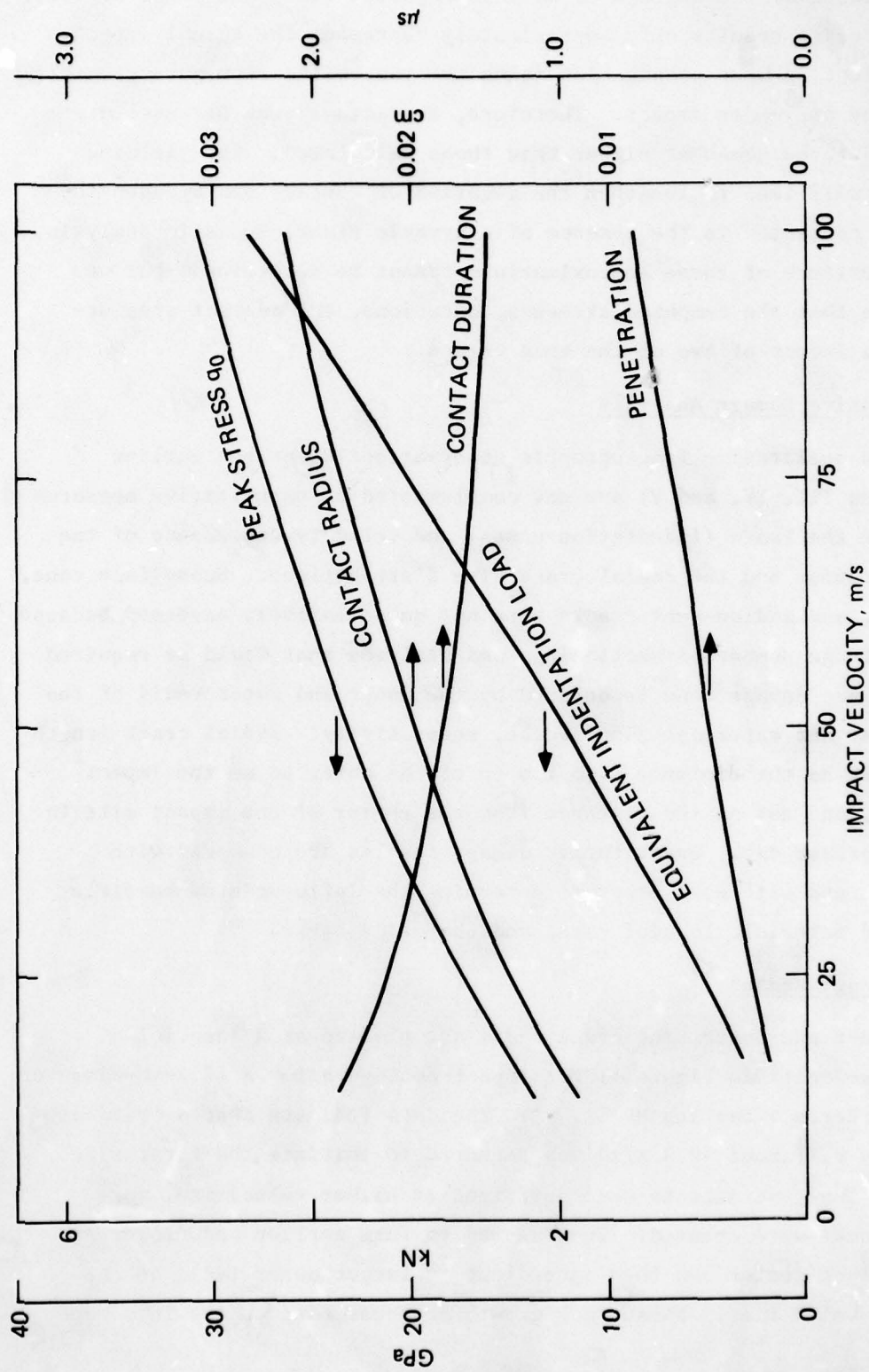
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FIGURE 38 DYNAMIC CONTACT PARAMETERS (Particle: 2.4-mm-Diameter Steel Sphere Target: HP Si_3N_4)



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FIGURE 39 DYNAMIC CONTACT PARAMETERS (Particle: 1.6-mm-Diameter WC Sphere Target: HP Si_3N_4)



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FIGURE 40 DYNAMIC CONTACT PARAMETERS (Particle: 1.6-mm-Diameter WC Sphere. Target: RB Si₃N₄)

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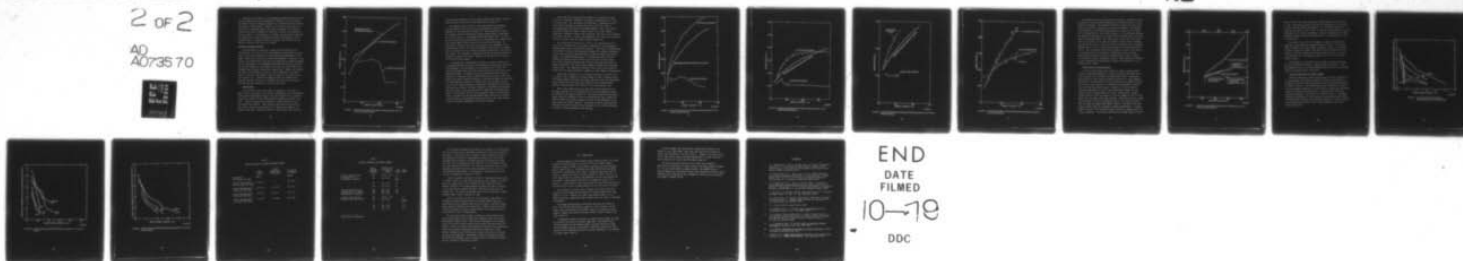
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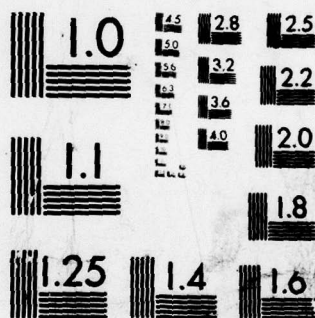
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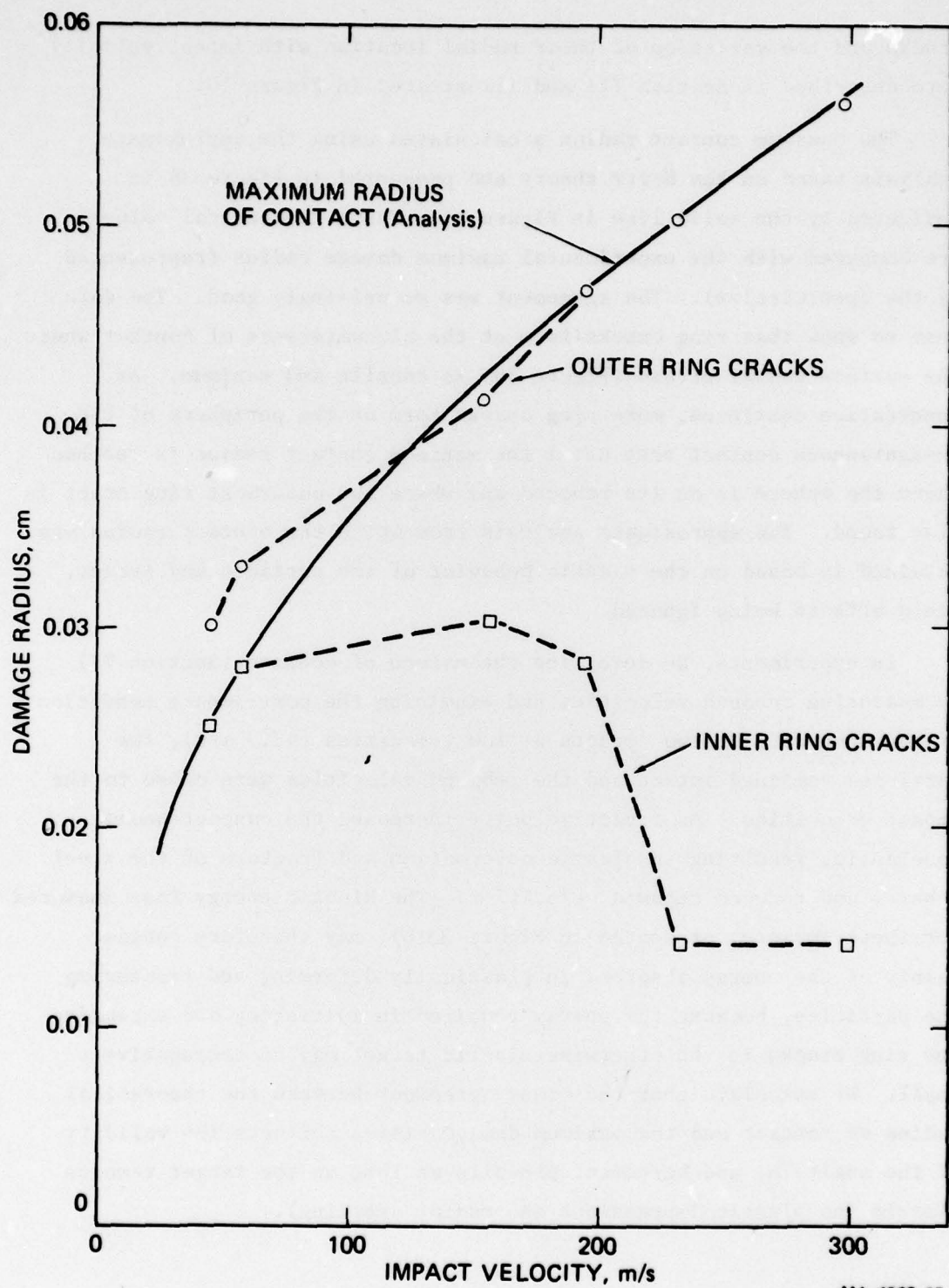
Because of the neglect of wave propagation times and yield effects, the preceding results only approximately represent the actual impact. The neglect of wave propagation makes the two bodies seem more flexible than they are under impact. Therefore, the actual peak stresses are expected to be somewhat higher than those calculated. The yielding process will tend to lengthen the duration of contact and broaden the area of contact. In the absence of a dynamic elastic-plastic analysis, the importance of these approximations cannot be determined, but we estimate that the computed stresses, durations, and contact area are within a factor of two of the true values.

Quantitative Damage Analysis

The qualitative fractographic observations described earlier (Sections III, IV, and V) are now complemented by quantitative measurements of the load- (indentation cases) and velocity-dependence of the damage radius and the radial crack size distributions. Subsurface cone, lateral, and median-vent cracks were not quantitatively assessed because of the large number of sectionings and analyses that would be required. The surface damage zone is defined by the inner and outer radii of the innermost and outermost ring cracks, respectively. Radial crack length was taken as the distance from tip to tip as observed on the impact surface, and not as the distance from the center of the impact site to its outermost tip. Quantitative damage results are compared with analysis and with each other to determine the influences of particle size and material, loading rate, and the oxide layer.

Damage Radius

Inner and outer ring crack radii are plotted as a function of impact velocity in Figure 41 for impact configuration A (2.4-mm-diameter steel spheres impacting HP Si_3N_4). The data indicate that a critical velocity V_1 (about 42.3 m/s) was required to initiate the first ring crack. Then, as impacts were performed at higher velocities, more ring cracks were created. They seemed to form earlier and closer to the contact center and then spread out to larger outer radii at the highest velocities. Subsurface growth of these ring cracks into cone



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FIGURE 41 DAMAGE RADIUS RESULTS FOR IMPACT CONFIGURATION A (2.4-mm-Diameter Steel Sphere/HP Si_3N_4)

cracks and the variation of their radial location with impact velocity were described in Section III and illustrated in Figure 10.

The maximum contact radius a calculated using the approximate analysis based on the Hertz theory and presented in Figure 38 is indicated by the solid line in Figure 41. These theoretical values are compared with the experimental maximum damage radius (represented by the open circles). The agreement was surprisingly good. The data seem to show that ring cracks form at the circumference of contact where the surface radial stress (Figure 36) is tensile and maximum. As penetration continues, more ring cracks form on the periphery of the instantaneous contact zone until the maximum contact radius is reached where the sphere is on its rebound and where the outermost ring crack is also found. The approximate analysis from which the contact radius was obtained is based on the elastic behavior of the particle and target, yield effects being ignored.

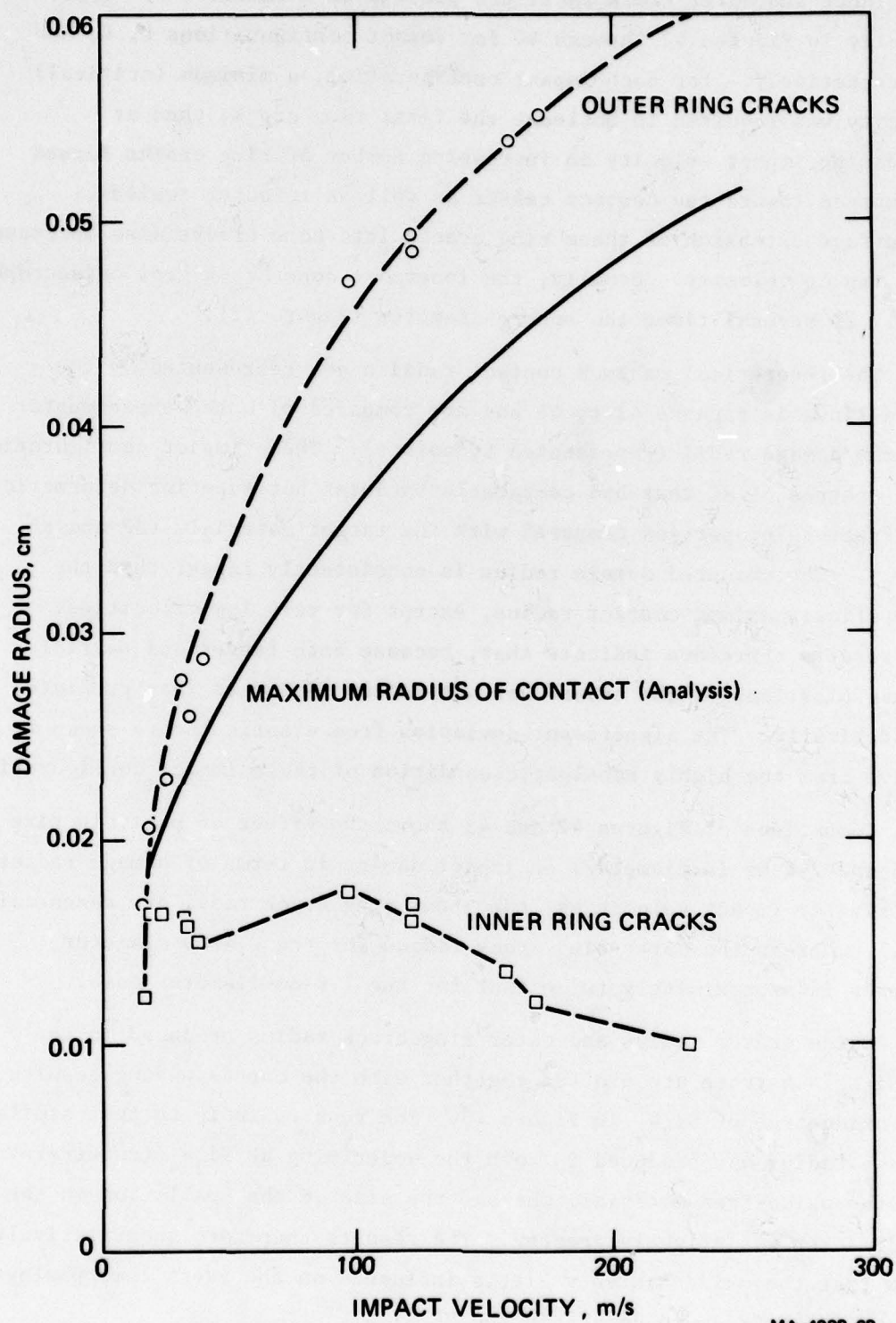
In experiments, we determine the nature of contact (Section VI) by measuring rebound velocities and examining the post-impact condition of the spheres. In two impacts at low velocities (42.3 m/s), the particles remained intact and the rebound velocities were close to the impact velocities. As impact velocity increased the contact became nonelastic, resulting in plastic deformation and fracture of the steel spheres and reduced rebound velocities. The kinetic energy loss measured for these impacts, presented in Figure 33(b), may therefore consist mainly of the energy absorbed in plastically deforming and fracturing the particles, because the energy required in initiating and extending the ring cracks in the otherwise elastic target may be comparatively small. We speculate that the close agreement between the theoretical radius of contact and the maximum damage radius reflects the validity of the analysis, and agreement prevails as long as the target remains elastic (no plastic impressions and radial cracking).

Inner and outer crack radii are plotted as a function of impact velocity in Figures 42 through 44 for impact configurations B, C, and D, respectively. For each impact configuration, a minimum (critical) velocity was required to nucleate the first ring crack, then at increasing impact velocity an increasing number of ring cracks formed and spread toward the contact center as well as to outer regions. Subsurface extension of these ring cracks into cone cracks also increased with impact velocity. Usually, the innermost cone crack grew catastrophically to several times the sphere diameter (Figure 12).

The theoretical maximum contact radii a are represented by the solid lines in Figures 42 to 44 and are compared with the experimental maximum damage radii (represented by points). These impact configurations used spheres of WC that had comparable hardness but superior deformation and fracture properties compared with the target materials (HP and RB Si_3N_4). The measured damage radius is consistently larger than the theoretical maximum contact radius, except for very low velocities. The results therefore indicate that, because both target and particle behave plastically, the actual contact radius is larger than predicted theoretically. The significant deviation from elastic theory seems to derive from the highly nonelastic condition of these impact configurations.

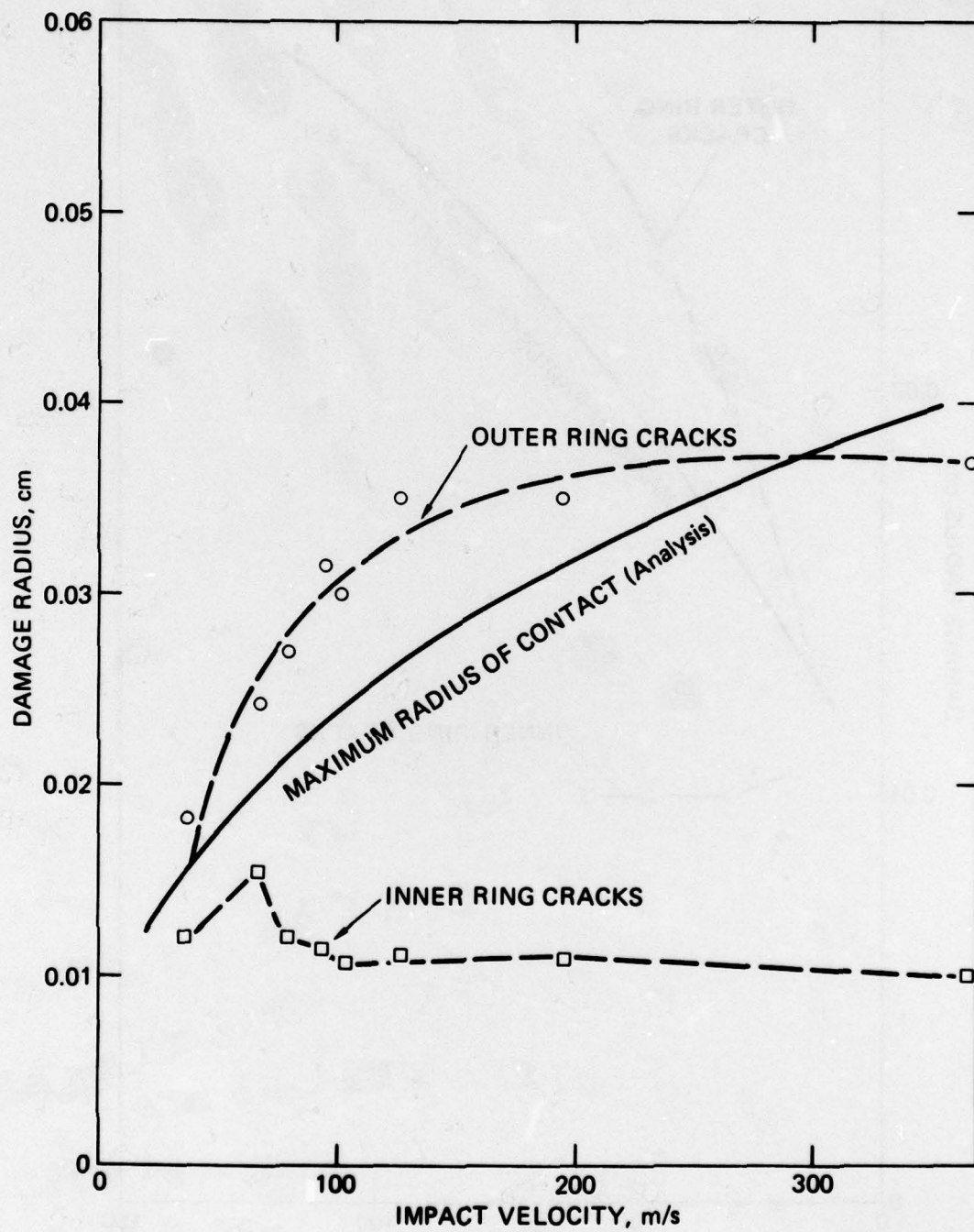
Comparison of Figures 42 and 43 shows the effect of particle size (1.6 and 2.4 mm in diameter) on impact damage in terms of damage radius. For similar impact velocities, the inner ring crack radii are essentially equal, whereas the outer ring crack radius for the 2.4-mm diameter spheres is approximately twice that for the 1.6-mm-diameter case.

Oxide crater radius and outer ring crack radius produced in the HP Si_3N_4 substrate are plotted together with the corresponding results for oxide-free HP Si_3N_4 in Figure 45. The results indicate that similar damage radius was produced in both the underlying HP Si_3N_4 (substrate) and the oxide-free material, whereas the size of the spallation in the oxide layer was slightly greater. The results therefore quantitatively show that the oxide has very little influence on the overall morphology or extent of fracture damage in the HP Si_3N_4 .



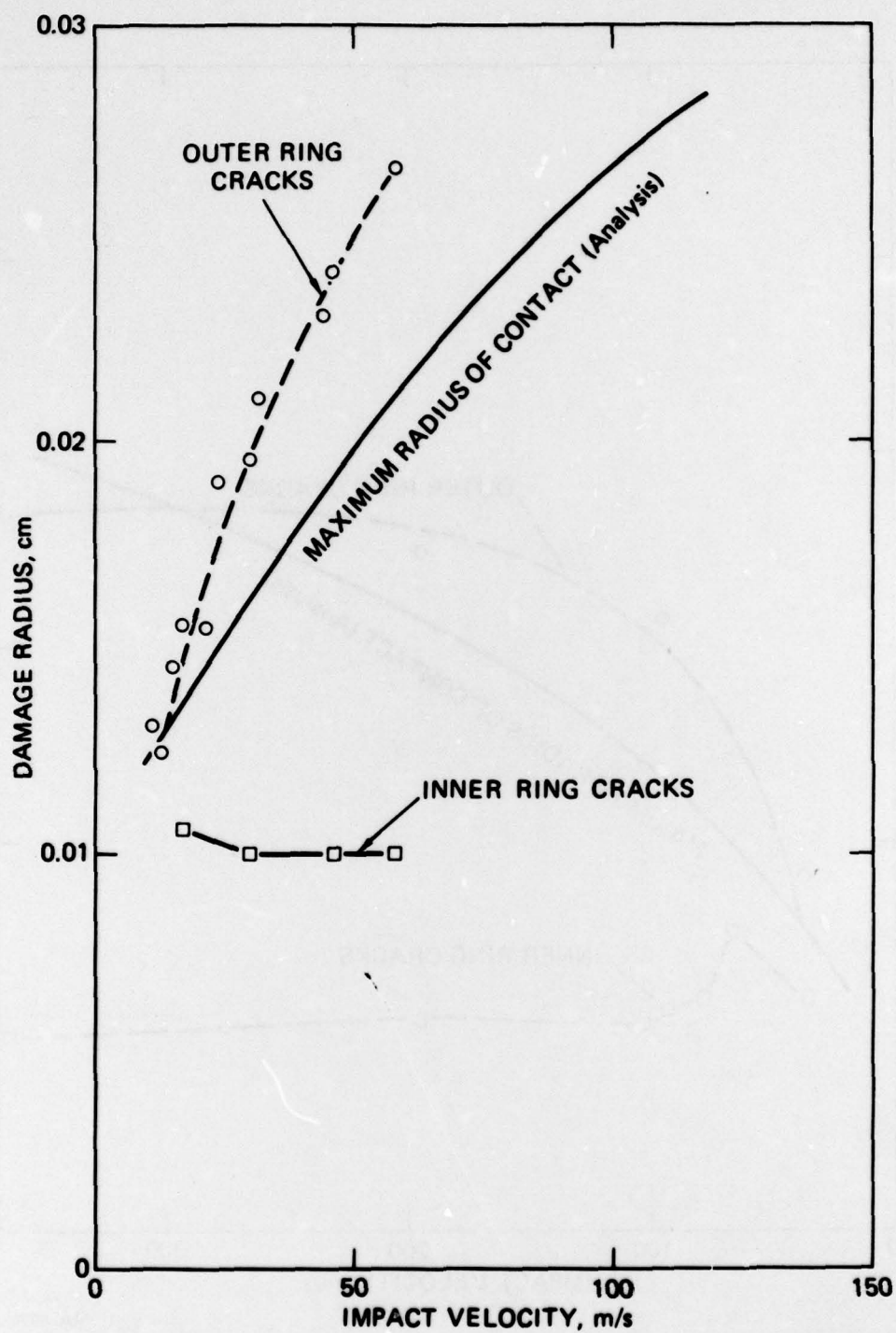
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FIGURE 42 DAMAGE RADIUS RESULTS FOR IMPACT CONFIGURATION B (2.4-mm-Diameter WC Sphere/HP Si_3N_4)



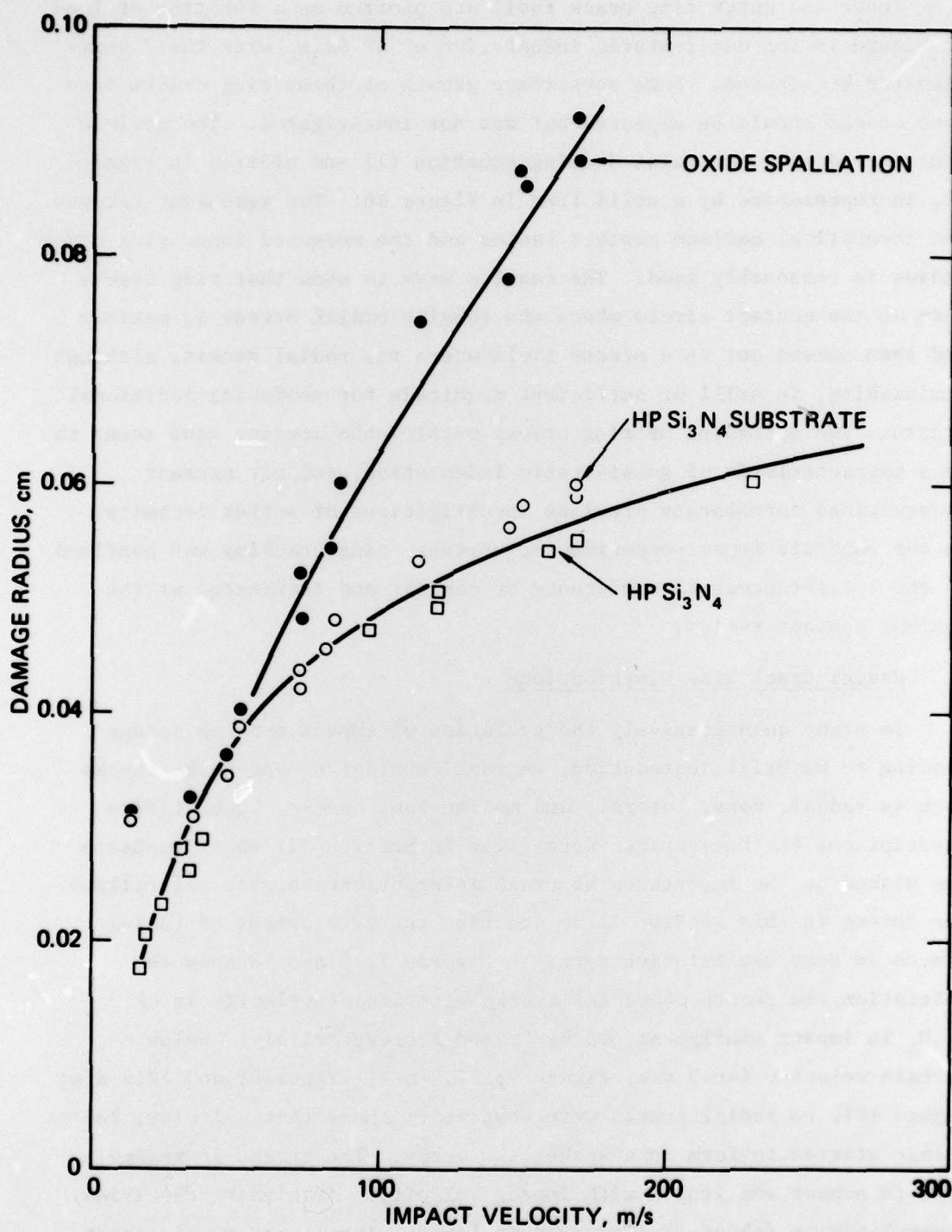
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FIGURE 43 DAMAGE RADIUS RESULTS FOR IMPACT CONFIGURATION C (1.6-mm-Diameter WC Sphere/HP Si_3N_4)



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FIGURE 44 DAMAGE RADIUS RESULTS FOR IMPACT CONFIGURATION D (1.6-mm-Diameter WC Sphere/RB Si₃N₄)



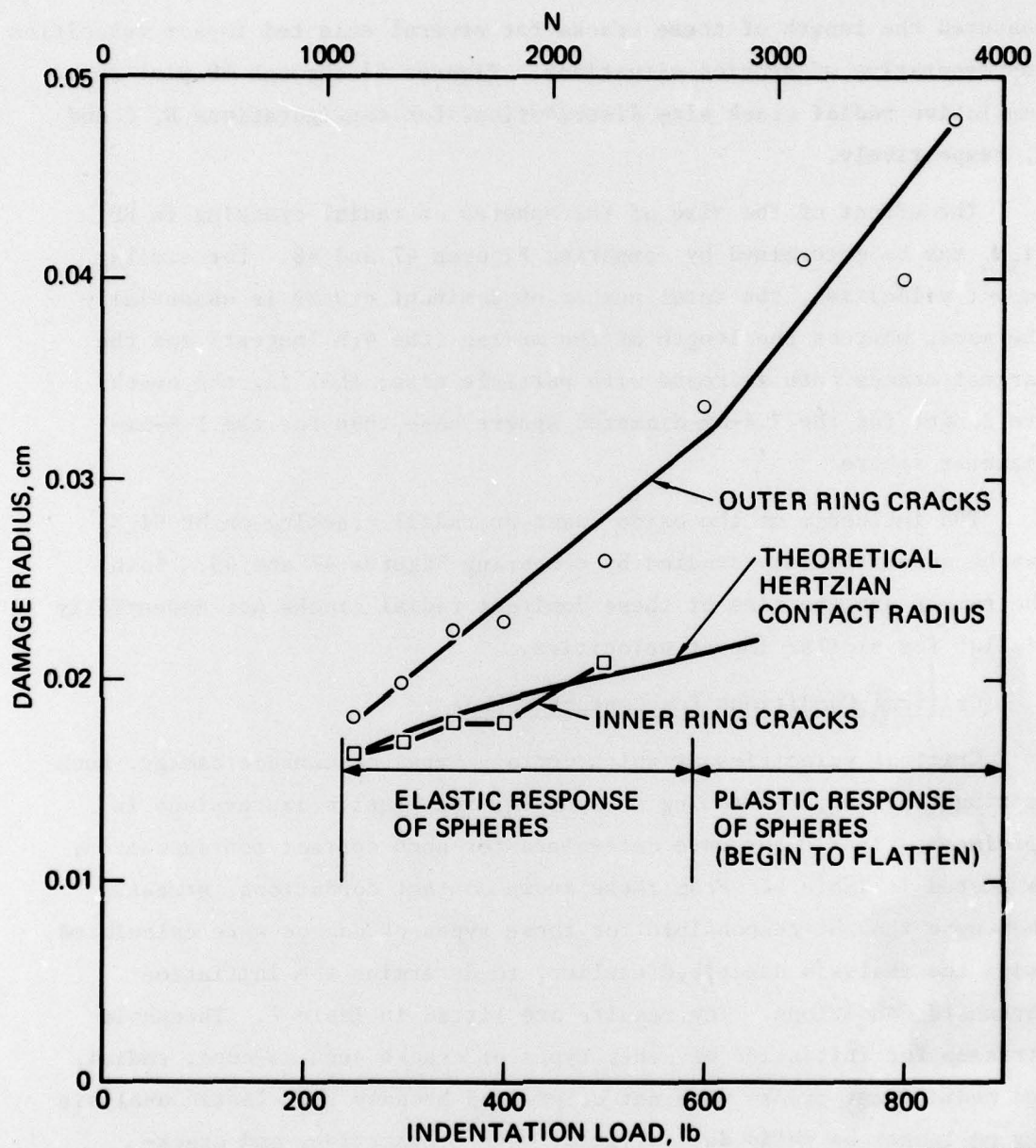
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FIGURE 45 DAMAGE RADIUS RESULTS FOR OXIDIZED AND UNOXIDIZED HP Si₃N₄ (2.4-mm-Diameter WC Spheres)

Inner and outer ring crack radii are plotted as a function of load in Figure 46 for quasi-static indentation of HP Si_3N_4 with the 2.4-mm-diameter WC spheres. Some subsurface growth of these ring cracks into cone cracks should be expected but was not investigated. The maximum contact radius a , calculated using equation (1) and plotted in Figure 35, is represented by a solid line in Figure 46. The agreement between the theoretical maximum contact radius and the measured inner ring crack radius is reasonably good. The results seem to show that ring cracks form on the contact circle where the tensile radial stress is maximum and then spread out in a stress field where the radial stress, although diminishing, is still of sufficient magnitude for producing additional cracks. The spreading of ring cracks outside the contact zone seems to be a characteristic of quasi-static indentation, and our present observations corroborate previous investigations of softer ceramics. In our particle impact experiments, however, ring cracking was confined to the instantaneous circumference of contact and terminated at the maximum contact radius.

Radial Crack Size Distributions

To study quantitatively the evolution of impact erosion damage leading to material degradation, we must consider second-stage cracks such as radial, cone, lateral, and median-vent cracks. Qualitative descriptions of these cracks were given in Section III where emphasis was placed on the importance of crack interactions to material failure. Our intent in this section is to describe the development of radial cracks in some quantitative detail. Figures 7, 8 and 18 show the initiation and growth of radial cracks with impact velocity in HP Si_3N_4 in impact configurations B, C, and F, respectively. Below a certain velocity (46.2 m/s, Figure 7; 67.7 m/s, Figure 8; and 72.6 m/s; Figure 18), no radial cracks were observed. Above that velocity, radial cracks started to form in a spoke-like array. The cracks increased both in number and length with impact velocity. At higher velocities, several cracks (about 9) increased in length significantly. A larger number (from 20 to 25) of shorter radial cracks also appeared near the contact periphery. The shorter cracks have an average length of about



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FIGURE 46 DAMAGE RADIUS RESULTS FOR QUASI-STATIC INDENTATION (2.4-mm-Diameter WC Spheres/HP Si_3N_4)

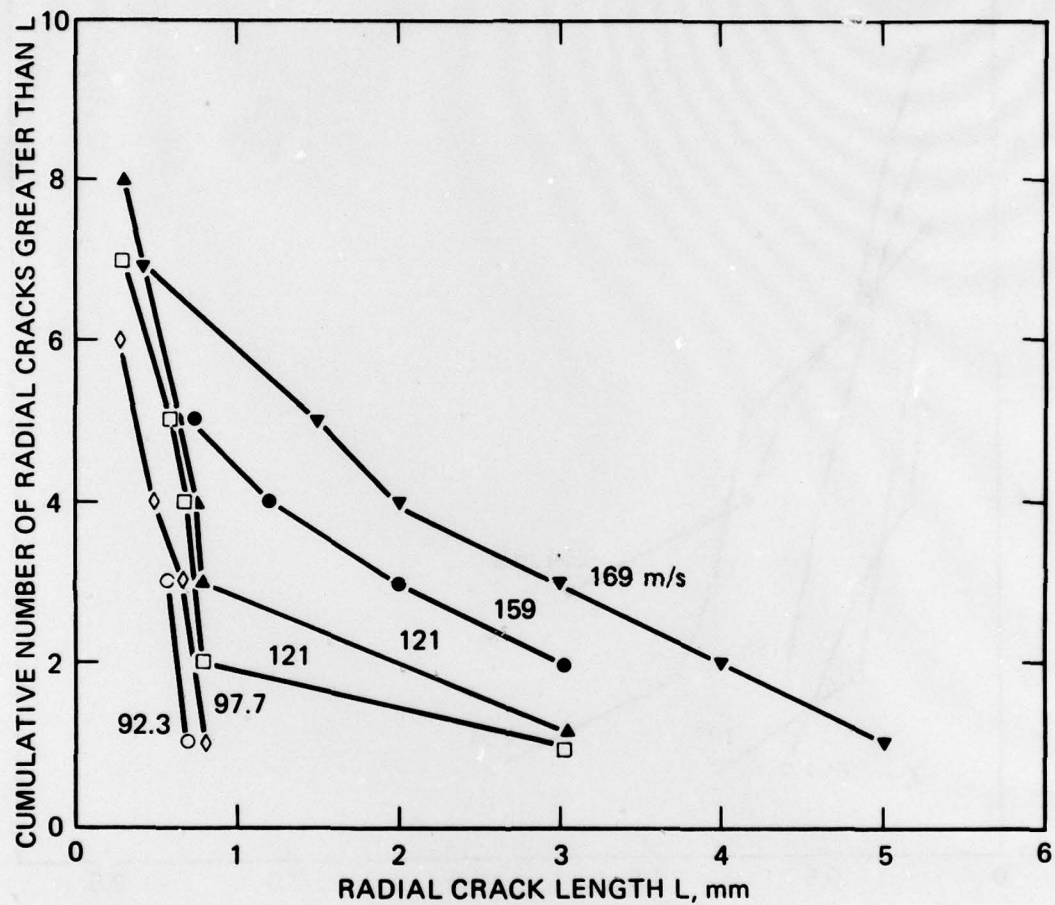
100 μm , whereas the larger cracks (now called dominant) grew as long as 5 mm. The dominant radial cracks should be important to the problem of impact-induced strength degradation. We counted the number and measured the length of these cracks for several selected impact velocities representative of erosion situations. Figures 47 through 49 plot these cumulative radial crack size distributions for configurations B, C and F, respectively.

The effect of the size of the spheres on radial cracking in HP Si_3N_4 may be determined by comparing Figures 47 and 48. For similar impact velocities, the total number of dominant cracks is essentially the same, whereas the length of the median (the 4th longest) and the largest cracks both increase with particle size; that is, the cracks are longer for the 2.4-mm-diameter sphere case than for the 1.6-mm-diameter sphere.

The influence of the oxide layer on radial cracking on HP Si_3N_4 may be quantitatively studied by comparing Figures 47 and 49. Both the number and the size of these dominant radial cracks are essentially similar for similar impact velocities.

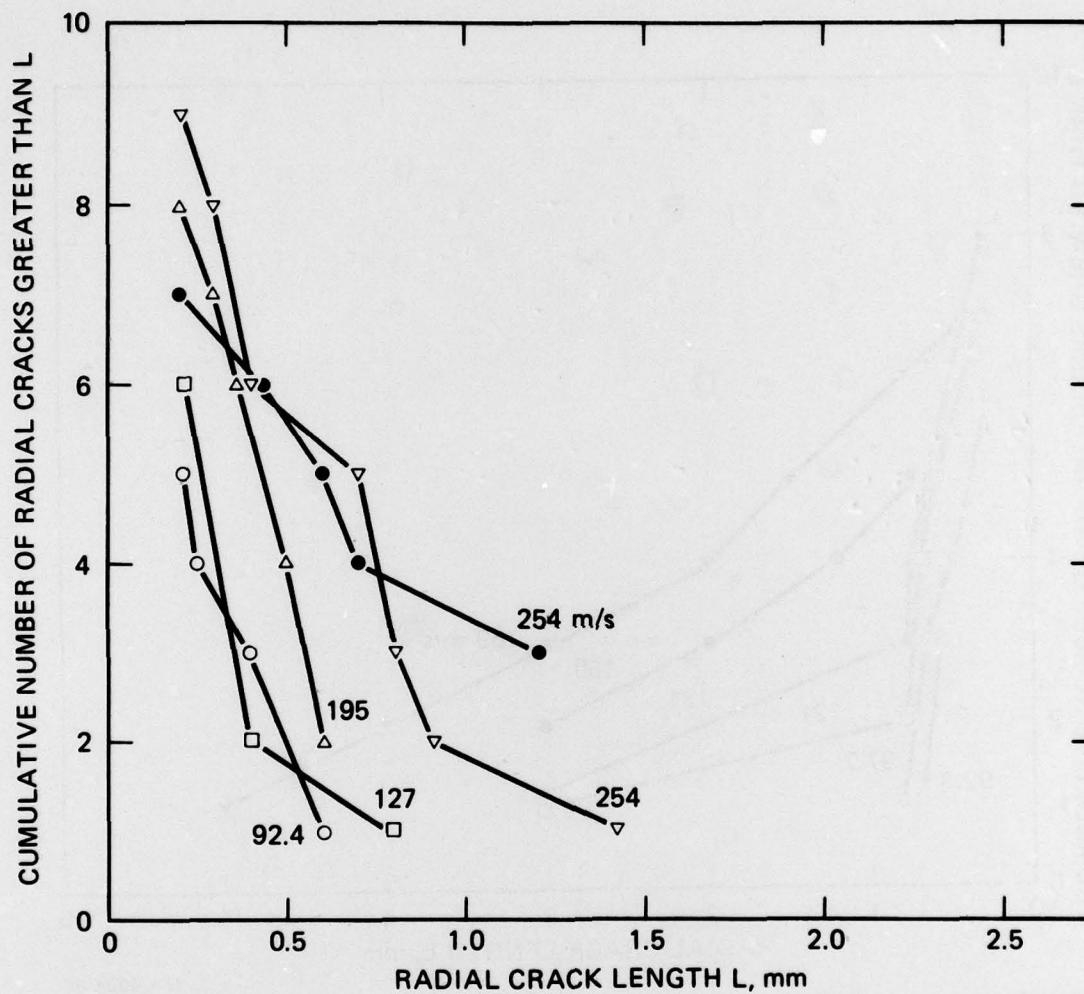
Critical Conditions for Contact Damage

Critical velocities at which certain types of contact damage, such as ring cracking, flattening of spheres, and plastic impressions in specimens, first occur were determined for each contact configuration, as listed in Table 6. From these known contact conditions, stresses that were thought responsible for these types of damage were calculated, using the analysis described earlier, to determine the initiation threshold conditions. The results are listed in Table 7. Threshold stresses for initiation of other types of cracks such as cone, radial, and median-vent cracks were not determined because the elastic analysis may no longer be valid due to yield, wave propagation, and crack-interaction effects.



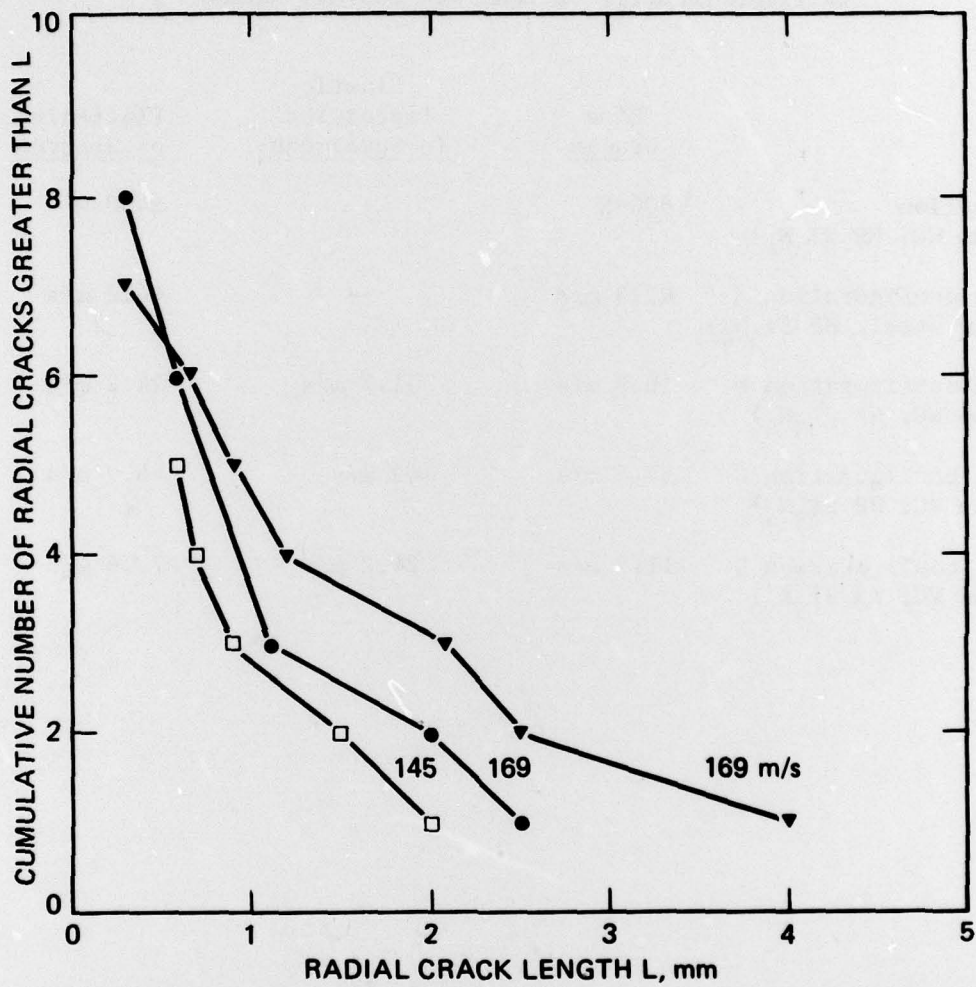
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FIGURE 47 RADIAL CRACK SIZE DISTRIBUTIONS
(Impact Configuration B: 2.4 mm WC/HP Si_3N_4)



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FIGURE 48 RADIAL CRACK SIZE DISTRIBUTIONS (Impact Configuration C: 1.6 mm WC/
HP Si_3N_4)



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FIGURE 49 RADIAL CRACK SIZE DISTRIBUTIONS (Impact Configuration F: 2.4 mm WC/
Oxidized HP Si_3N_4)

Table 6

CRITICAL VELOCITY OR LOAD FOR CONTACT DAMAGE

	<u>Ring Cracks</u>	<u>Plastic Impression in Specimens</u>	<u>Flattening of Spheres</u>
Indentation (2.4-mm WC, HP Si_3N_4)	800 N	--	2450 N
Impact configuration A (2.4-mm steel, HP Si_3N_4)	42.3 m/s	--	46.2 m/s
Impact configuration B (2.4-mm WC, HP Si_3N_4)	16.9 m/s	31.7 m/s	24.2 m/s
Impact configuration C (1.6-mm WC, HP Si_3N_4)	37.6 m/s	~ 42 m/s	46.2 m/s
Impact configuration D (1.6-mm WC, RB Si_3N_4)	11.3 m/s	24.2 m/s	> 72.6 m/s

Table 7

CRITICAL STRESSES FOR CONTACT DAMAGE

	<u>Si₃N₄ Specimen Material</u>	<u>Sphere Type and Diameter (mm)</u>	<u>σ_{zz} (GPa)</u>	<u>σ_{rr} (GPa)</u>
Critical normal stress (compressive) for flattening of spheres	HP	Steel, 2.40	17	
	HP	WC, 2.40	25 (26*)	
	HP	WC, 1.60	31	
	RB	WC, 1.60	27	
Critical normal stress (compressive) for plastic impressions in specimens	HP	WC, 2.40	28	
	HP	WC, 1.60	30	
	RB	WC, 1.60	17	
Critical radial stress (tensile) for ring cracks	HP	Steel, 2.40		3.1
	HP	WC, 2.40		2.8 (2.9*)
	HP	WC, 1.60		2.9
	RB	WC, 1.60		2.0

*Quasi-static indentation.

The incipient innermost ring cracks on the surface of the specimens give the location and the threshold condition for ring-crack nucleation and growth. The tensile radial stress at these locations represents the threshold and should be approximately constant for each contact configuration irrespective of impact velocity (or indentation load). For each contact configuration, the variation of the threshold stress for ring cracking with velocity (or load) was reasonably small ($\pm 10\%$), seemingly indicating that the elastic analysis is valid at least in the incipient damage regime. The average of these stresses for each contact configuration is listed in Table 7.

Contact strengths (expressed in terms of compressive normal stress at contact center) of steel and WC spheres are also compared in Table 7. Steel has significantly lower strength (17 GPa) than WC (25 GPa). Dynamic strength of the 24-mm-diameter WC spheres (25 GPa) is similar to its quasi-static strength (26 GPa), indicating little effect of loading rate. WC spheres of smaller size (1.6 mm in diameter) exhibit somewhat higher contact strength (31 GPa).

Critical stresses required for creating plastic impressions in different specimen materials show that reaction bonded Si_3N_4 has significantly lower strength than hot pressed Si_3N_4 . Reaction bonded Si_3N_4 similarly exhibits lower resistance to ring cracking, and there is a close agreement between strength values for HP Si_3N_4 determined from different contact configurations.

The calculated radial tensile stresses for ring cracking in HP Si_3N_4 and RB Si_3N_4 are much higher than handbook values for flexural and uniaxial tensile stress. This may be reasonable for two reasons. First, the critical radial tensile stresses were calculated based on elastic contact, but plastic flow beneath the particle may significantly reduce the stresses considerably below these theoretical values. Second, stresses at the location where ring cracks form are triaxial and have steep gradients. Comparison of material strengths obtained from different loadings, such as particle impact and uniaxial tension may not, therefore, be entirely correct.

VIII CONCLUSIONS

Fracture damage in fully dense HP Si_3N_4 caused by impact of 2.4-mm-diameter WC spheres begin at a velocity of 17 m/s (radial tensile stresses of about 2.8 GPa) in the form of shallow ring cracks (Figure 7 and 34, Table 6 and 7). Increasing velocities produce more and deeper ring cracks, and Hertzian cone cracks. A plastic impression forms at 32 m/s (a compressive stress of 28 GPa, Figure 34, Table 6 and 7); at still higher velocities lateral and median-vent cracks are produced and the number of large radials increases to a maximum of about 8 or 9 (Figure 7). Interaction of these crack types with each other and with the specimen surface occurs at approximately 231 m/s and results in erosion.

The presence of an oxide scale (roughly 20 μm thick) had little influence on the morphology and extent of fracture damage in HP Si_3N_4 (pages 37 to 45). However, the oxide layer spalled easily at low impact velocities, suggesting higher erosion rates for Si_3N_4 in oxidizing environments.

The damage phenomenology in 85% dense, RB Si_3N_4 was similar to that in the hot-pressed material (pages 30 to 36). However, for RB Si_3N_4 , ring cracking and all subsequent stages of the damage process began at roughly two-thirds the stress needed to produce damage in HP Si_3N_4 (Table 7).

A significant effect of particle size was observed (pages 30, 100), but loading rate effects were small (page 106). Decreasing the WC particle diameter by 33% caused roughly a two-fold increase in ring crack threshold velocity (no increase in threshold stress) and a 50% increase in the threshold velocity for plastic impression (a 7% increase in stress, Table 6 and 7).

Fracture damage under quasi-static loading rates appeared to be similar to that under impact loads, and ring cracks were observed at similar load thresholds (~ 2.8 GPa, Table 7). However, flow and fracture of the indenting WC spheres prevented application of higher loads and hence production of further specimen damage (page 51).

An elastic Hertzian analysis of the stress field adequately predicted our observations of ring cracking in HP Si_3N_4 (page 92, Figure 41). In the elastic-plastic regime, however, the measured area of fracture damage was consistently broader than predicted by elastic theory, indicating that yielding and wave propagation effects must be accounted for (page 93, Figures 42-44).

REFERENCES

1. D. A. Shockey, K. C. Dao, L. Seaman, and D. R. Curran, "Nucleation and Growth of Cracks in CVD ZnS under Particle Impact," Annual Report Part II to the Office of Naval Research, Arlington, VA, on Contract N00014-76-C-0657 (1979).
2. D. D. Cubicciotti, R. L. Jones, and K. H. Lau, "High Temperature Oxidation and Mechanical Properties of Silicon Nitride," Interim Report 5522-1 to the Air Force Office of Scientific Research, Bolling AFB, D.C., on Contract F44620-76-C-0016 (1977).
3. D. D. Cubicciotti, R. L. Jones, K. H. Lau, and D. J. Rowcliffe, "High Temperature Oxidation and Mechanical Properties of Silicon Nitride," Interim Report 5522-2 to the Air Force Office of Scientific Research, Bolling AFB, D.C., on Contract F44620-76-C-0016 (1978).
4. B. Lawn and T. R. Wilshaw, "Review Indentation Fracture: Principles and Applications," *J. Mat. Sci.* 10, 1049-1081 (1975).
5. A. G. Evans and T. R. Wilshaw, "Quasi-Static Solid Particle Damage in Brittle Solids - I. Observations, Analysis and Implications," *Acta Metallurgica* 24, 939-956 (1976).
6. A. G. Evans, private communication (1979).
7. S. Timoshenko and J. N. Goodier, Theory of Elasticity 2nd Ed., (McGraw-Hill Book Co., Inc., New York, 1951).
8. G. N. Bycroft, "Forced Vibrations of a Rigid Circular Plate on a Semi-Infinite Elastic Space and on an Elastic Stratum," *Philosophical Transactions of the Royal Society of London, Series A*, No. 948, pp. 327-368 (1956).
9. I. S. Gradshteyn and I. W. Ryzhik, Table of Integrals, Series, and Products (Academic Press, New York, 1965).
10. G. N. Watson, A Treatise on the Theory of Bessel Functions, 2nd Ed. (Cambridge University Press 1944).
11. A Erdelyi, Ed., Higher Transcendental Functions, Bateman Manuscript Project, Vol. I (McGraw-Hill Book Co., Inc., New York, 1953).